PNWD-3035
BNFL-RPT-027 Rev. 0

Combined Entrained Solids and Sr/TRU Removal from AN-107 Diluted Feed

R. T. Hallen
P. R. Bredt
K. P. Brooks
L. K. Jagoda

August 2000

Prepared for BNFL, Inc.
under Contract W375-LC-98-4168

PNWD-3035

LEGAL NOTICE

PNWD-3035
BNFL-RPT-027 Rev. 0

Combined Entrained Solids and Sr/TRU Removal from AN-107 Diluted Feed

R. T. Hallen
P. R. Bredt
K. P. Brooks
L. K. Jagoda

August 2000

Prepared for
BNFL, Inc.
under Contract W375-LC-98-4168

Battelle, Pacific Northwest Division
Richland, Washington 99352

SUMMARY

Waste from Hanford underground storage tank 241-AN-107 is a candidate low-activity waste (LAW) for Envelope C. Envelope C wastes require pretreatment to remove radioactive Sr and TRU (along with cesium and technetium) before immobilization. The baseline pretreatment process planned for Sr/TRU removal was precipitation with added strontium and iron. However, studies have shown that the Sr/Fe precipitates were very difficult to filter. An alternative treatment being evaluated uses permanganate instead of iron. Permanganate treatment has been shown to be effective for decontaminating waste from Hanford Tank SY-101.

Battelle conducted small-scale experiments with archived AN-107 waste over a period of about three months to determine the effectiveness of the permanganate treatment process. These tests showed that permanganate treatment alone would provide adequate TRU removal, however, permanganate alone would not provide adequate Sr removal. The preferred Sr/TRU removal process involved addition of strontium and permanganate. Test conditions that provided adequate Sr/TRU decontamination were identified. These test conditions were further evaluated with a 1-L batch of archived AN-107, which provided a large enough volume of waste to conduct crossflow filtration studies. These tests showed that Sr/TRU removal could be accomplished by addition of a strontium solution followed by permanganate solution. The resulting precipitate could be removed effectively by crossflow filtration.

The original target sodium concentration for AN-107 diluted feed was 7.7M. The waste was further diluted and additional caustic added before Sr/TRU precipitation. The target concentrations for the treated waste were 6.0M sodium, 1.0M free hydroxide, 0.075M strontium, and 0.05M permanganate. Approximately 1.4-L of AN-107 diluted feed were treated. Decontamination of strontium-90 and TRU (Am-241) in the supernatant was greater than needed to meet the immobilized low-activity waste (ILAW) requirements (less than 100 nCi/g TRU and less than 20 Ci/m^3 Sr-90 in the final ILAW). The stronium-90 decontamination factor (DF) was consistently greater than 50 and the Am-241 DF greater than 25. The target DFs were 10 for Sr-90 and 5 for Am-241. These DFs include the contribution from the removal of the entrained solids although this was relatively small. The removal of the entrained solids accounted for about 8% of the DF for Sr-90 and about 17% of the DF for the alpha emitters.

Crossflow filtration tests with archived AN-107 showed that the entrained solids could not be readily removed from AN-107 waste prior to Sr/TRU treatment. However, the filterability, as determined by filter flux rate, increased by an order of magnitude after the treatment process. For AN-107 diluted feed, crossflow filtration tests were conducted in the Cell Unit Filter (CUF) system with the Sr/TRU precipitated waste only. Results showed that the treated waste could be effectively filtered by crossflow filtration. A parametric study was conducted with relatively low, 1.9 wt%, initial solids loading of the treated waste. At target transmembrane pressure (TMP) of 50 psi and crossflow velocity of 12.2ft/sec, the average flux rate was 0.03 gpm/ft^2 or 1.75 m^3/m^2/day and the permeability was 0.53 m/day/bar. The treated waste slurry was dewatered in the CUF to approximately 4 wt%. Additional dewatering in the CUF was not possible because the minimum operating volume of the CUF was reached. Solids' washing was not conducted in the CUF because of the low solid loading in the dewatered slurry. Additional filtration tests will need to be conducted to determine maximum solids loading.

TERMS AND ABBREVIATIONS

AEA	alpha energy analysis
BNFL	BNFL, Inc; subsidiary of British Nuclear Fuels, Ltd.
DF	decontamination factor
DI water	deionized water
EQL	estimated quantitation level
GEA	gamma energy analysis
ICP	inductively coupled plasma/atomic emission spectrometry
MDL	method detection limit
MRQ	minimum reportable quantity
RPD	relative percent difference
SAL	Shielded Analytical Laboratory

UNITS

°C	degrees Centigrade
ft/s	feet per second
g	gram
g/mL	gram per milliliter
µg/g - µg/mL	microgram per gram / microgram per milliliter
µCi/g - µCi/mL	microcurie per gram / microcurie per milliliter
M	mole/liter
mL	milliliter
mmole/mL	millimole per milliliter
nCi/g	nanocurie per gram
pCi/g	picocurie per gram
Vol%	volume percent
Wt%	weight percent

CONTENTS

FIGURES

TABLES

1.0 INTRODUCTION

BNFL Inc. was awarded the Privatization Contract for treatment of Hanford underground storage tank wastes as part of the River Protection Project-Waste Treatment Plant (RPP-WTP). In Part B-1, Battelle is conducting technology development and demonstration of process flowsheet steps. Three candidate low-activity waste types have been identified, Envelope A, Envelope B and Envelope C. Treatment and disposal of the liquid (supernatant) fraction of Envelope C wastes, such as tank 241-AN-107, requires that transuranics (TRU) and radioactive strontium be removed. Because of the high concentration of organic complexants in this waste (Complexant Concentrate waste), conventional separation processes (e.g., ion exchange) are not effective.

During Part A-1 of privatization, Savannah River Technology Center (SRTC) developed a Sr/TRU removal process which involved isotopic dilution and precipitation with added strontium and iron (SRTC 1997a-d). While this treatment process provided the necessary supernatant decontamination, the resulting precipitate could not be filtered. The search began for an alternate treatment process. Battelle proposed permanganate be examined as an alternative, because it had been demonstrated to work with waste from Hanford tank SY-101, which also contained high levels of organic complexants (Orth et al. 1995).

Permanganate has been examined as an oxidant (decomplexing waste (Orth et al. 1995), solubilizing chromium (Rapko et al. 1995, Rapko 1998), and oxidation of technetium species to pertechnetate (Schroeder)) for treating tank wastes. Permanganate was found to oxidize chromium first, then organic carbon, and lastly nitrite. For wastes such as Tank SY-101, the chromium in the sludge consumes as much as half the permanganate. Orth et al. recommended permanganate doses of 0.1M for decomplexing SY-101 type wastes. At this level of permanganate, decontamination factors (DF) of > 143 were obtained for Sr and 28.5 for Pu. AN-107 does not have the high chromium values in the sludge so permanganate is expected to be effective at lower concentrations.

Permanganate is also used as a precursor to MnO_2 and/or $Mn(OH)_2$ coprecipitants via the "Method of Appearing Reagents" (Krot et al. 1996). The method of appearing reagents requires the addition of a reductant to the waste to be treated. However, for Hanford wastes this is not necessary because reductants are already present in the waste. The resulting solids are effective coprecipitants for Pu and other TRU elements but generally not as effective as iron precipitates. Decontamination factors of greater 100 have been reported for various simulated waste streams.

The treatment scheme for Sr/TRU removal was developed from tests conducted at Battelle with waste simulants and actual waste (Hallen et al. 2000a). The final test conditions were defined by BNFL in a Test Specification (Townson 1999) document based on previous results. This test specification was used to prepare a general test plan for Sr/TRU removal tests. A test instruction was prepared which detailed the specifics for conducting this test with AN-107 diluted feed. The test instruction was used to record the specific details of the test, and is attached in Appendix A.

The proposed pretreatment flowsheet shows entrained solids are removed from the double-shell tank wastes. The entrained solids may be returned to BNFL as HLW for vitrification or as LAW for pretreatment depending on composition. Battelle used a Cell Unit Filtration System (CUF) equipped with a 0.1-µm filter element (Brooks et al. 1999) to conduct filtration tests with an archived AN-107 waste sample. These tests demonstrated that the entrained solids present in this waste could not easily be removed by crossflow filtration (Hallen et al. 2000b). For entrained solids removal, the initial flux dropped in less than a minute to 0.023 gpm/ft^2 and within 5 minutes had dropped to 0.0074 gpm/ft^2 at 55-psid transmembrane pressure (TMP) and 12.2 ft/s crossflow velocity. To prevent further plugging of the filter, no further testing was conducted at this condition. An attempt was made to collect sufficient filtrate to backpulse (clean) the filter. Only a small quantity of material could be collected in the backpulse chamber and two short backpulses were performed. A second condition was then tested at 70 psi and 9.3 ft/s. In this case, after 1 min the filtrate flux was 0.0079 gpm/ft^2. Testing was stopped at this point and entrained solids removal was determined to be not feasible for AN-107 waste.

The waste was drained from the CUF and Sr/TRU precipitation conducted on the waste with the entrained solids present. Approximately 75 mL of 1 M $Sr(NO_3)_2$ and 50 mL of 1M $NaMnO_4$ were added to 882 mL of the caustic adjusted (1M) waste drained from the CUF. The precipitated waste was digested at 50°C for 4 hours. The resulting slurry was cooled and transferred back to the CUF and filtration tests conducted. Figure 1.1 shows filtrate flux data for entrained solids removal and Sr/TRU precipitate removal from archived AN-107 at 55 psi TMP and 12.2 ft/s crossflow velocity. The filtrate flux was an order of magnitude higher for the treated waste, 0.11 gpm/ft^2 averaged over the hour of testing.

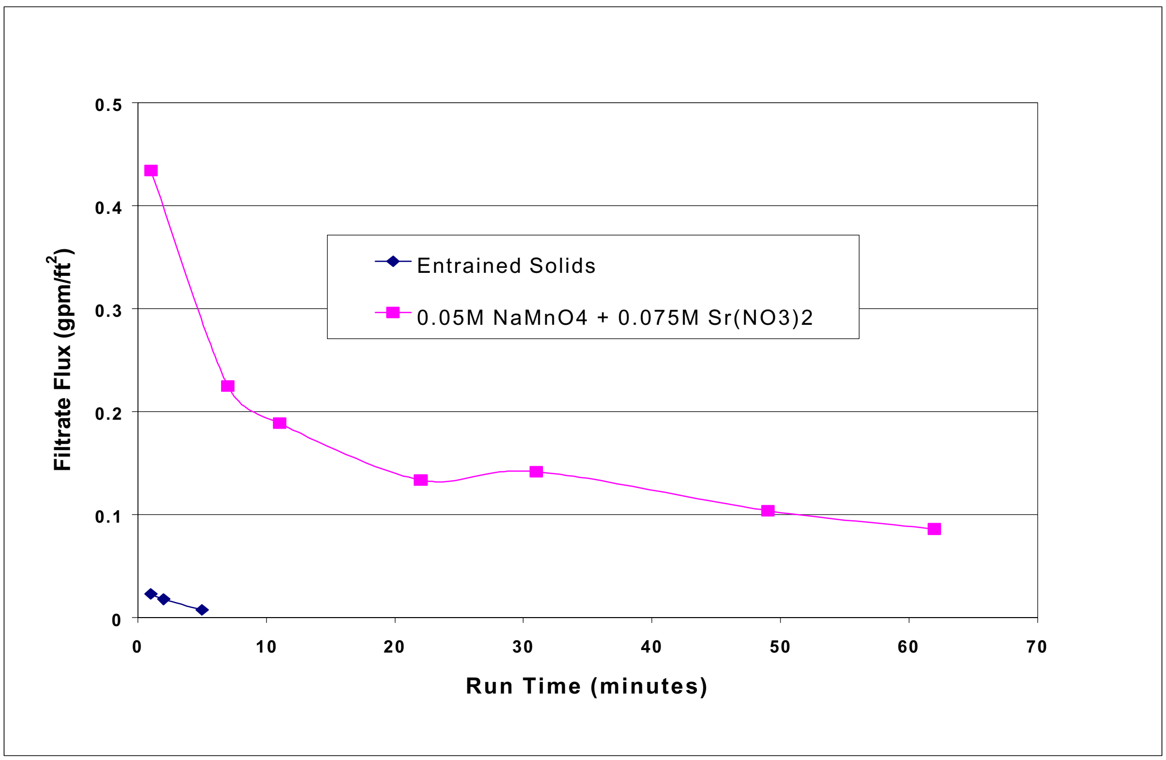


Figure 1.1. Comparison the Filtrate Flux for Entrained Solids Removal and Sr/TRU Precipitated Archived AN-107 Waste at 55 psi TMP and 12.2 ft/s Crossflow Velocity

Entrained solids removal from AN-107 diluted feed is expected to be even more difficult than from the archived waste. The archived waste was more dilute, suspended solids had been removed by settling, and the waste had been run through a cesium ion exchange column (Hendrickson 1997). Since the Sr/TRU precipitated waste with entrained solids are filterable and the entrained solids alone are not, the two filtration steps were combined. Furthermore, analysis of the entrained solids in the AN-107 diluted feed showed that they would be classified as HLW similar to the Sr/TRU precipitate and thus need not be sent back to the DOE for HLW storage prior to vitrification.

This report contains the results of Sr/TRU removal testing conducted at Battelle with AN-107 diluted feed. Test conditions and experimental procedures are described in Section 2.0. Results from waste treated with added Sr and permanganate are described in regards to Sr/TRU decontamination, chemical composition, solids removal, and physical and rheological properties of the waste in Section 3.0. The major conclusion and recommendations that evolved from this work are given in Section 4.0. The appendices contain the test instruction, data sheets, logbook entries, analytical data, rheograms, calculation, and staff role/responsibilities for this work.

2.0 TEST CONDITIONS AND EXPERIMENTAL PROCEDURES

The conditions for conducting the Sr/TRU removal tests were detailed in Sr/TRU Precipitation and Ultrafiltration Test Specification (Townson 1999) issued by BNFL. The Test Specification was used to prepare a Test Plan (TP 29953-013) that described the general requirements for the Sr/TRU removal tests to be conducted at Battelle. The actual test was conducted in accordance with Test Instruction-29953-052, which was specific to the Sr/TRU Removal test described in this report for AN-107 diluted feed. No deviations from the test instruction were necessary.

2.1 Description of Diluted Feed

A total of 17 samples from Tank AN-107 were received from Hanford's 222-S Laboratory between September 14 and 25, 1998. These samples were then composited and homogenized in a 4-L, glass kettle. The homogenized waste was sampled and characterized to represent the waste, as it exists in Tank AN-107 (Urie et al. 1999a). The composite waste was diluted to represent process flowsheet conditions, i.e., diluted feed. The diluted feed target sodium concentration was 7.7 M and the free hydroxide was 1.1 M. These compositing and dilution activities were conducted under Test Plan 29953-1.

The amount of sodium in the AN-107 diluted feed sample from the original, as-received waste is needed to determine the waste loading in the ILAW. The sodium concentration in the as-received AN-107 was 9.26M. The as-received waste (1.57-L) was combined with 0.16-L of decanted supernatant (9.0M Na) to give 1.73-L of waste with a sodium concentration of 9.2M. The diluted feed was prepared by adding 0.13-L of 19M NaOH and 0.44-L of 0.1M NaOH to the 1.73-L of waste. The AN-107 diluted feed had a calculated sodium concentration of 8.04M and 86.4 mole or weight percent of the sodium was from the as-received waste. The diluted feed was 75-volume percent as-received waste.

The AN-107 diluted feed was characterized as two individual components, supernatant and centrifuged solids (Urie et al. 1999b). The data from each component was used to calculate the starting composition of the diluted feed. The data from the individual samples were averaged, and the density was used for the supernatant, along with the percent centrifuged solids data to calculate the initial composition of the diluted feed. The calculated composition of the starting AN-107 diluted feed is shown in Table 2.1. The sodium concentration reported for the supernatant fraction, 7.5M, appears much lower than the expected 8.04M. However, the analyses conducted on the treated waste samples, reported in Section 3.0 of this report, also suggest the sodium concentration reported in Table 2.1 is too low. The sodium concentration of the diluted feed should therefore be close to the calculated sodium concentration of 8.04M.

Table 2.1. Calculated Composition of AN-107 Diluted Feed Before Treatment

BNFL ICP Analyte List	Supernatant Average (µg/mL)	Solids Average (µg/g)	Calculated Composition (µg/g)
Al	3930	7450	3120
Ba	4	46	5
Ca	439	368	323
Cd	47	34	35
Cr	146	716	138
Cu	21	20	16
Fe	1140	8900	1250
K	1270	659	1240
La	23	110	22
Mg	-	30	2
Mn	107	4990	329
Na	173500	136500	128000
Ni	392	277	288
Pb	256	770	218
Zn	19	64	17
Zr	43	202	40

Other ICP Analytes	Supernate Average (µg/mL)	Solids Average (µg/g)	Calculated Composition (µg/g)
As	95.5	80	70.72
Ce	27	215.5	29.83
Nd	70.5	318.5	65.44
P	496.5	427	368.23
Sr	2.6	6.3	2.79
Y	11	31.5	9.28

Rad. Chem. Analytes	Supernatant Average (µCi/mL)	Solids Average (µCi/g)	Calculated Composition (µCi/g)
Co-60 (GEA)	0.113	<	7.89E-02
Sr-90	75.9	191.5	6.27E+01
Cs-137 (GEA)	255.5	165	1.87E+02
Eu-154 (GEA)	0.6115	1.305	4.93E-01
Eu-155 (GEA)	0.3555	0.818	2.90E-01
Pu-238	0.00769	0.0438	7.60E-03
Pu-239+Pu-240	0.0314	0.1505	2.96E-02
Am-241 (GEA)	0.398	2.085	3.84E-01
Am-241 (AEA)	0.3785	1.47	3.39E-01
Cm-242	0.00144	0.00421	1.22E-03

Cm-243+Cm-244	0.01225	0.03095	1.01E-02
Total Beta	450	514.5	3.40E+02
Total Alpha	0.4465	1.83	4.05E-01
Alpha Sum	0.432	1.7	3.88E-01
	(µg/mL)	**(µg/g)**	**(µg/g)**
Total Cs	12	7.675	8.76E+00
Total U	73.1	103	5.63E+01

Analyte	**Supernatant Average (µg/mL)**	**Solids Average (µg/g)**	**Calculated Composition (µg/g)**
Tc-99 (ICP/MS)	4.315	3.77	3.20
TIC	16300	17850	12000
TOC	29900	32000	22500
TC (sum)	46200	50800	34800
Fluoride	6350	4400	4660
Chloride	1400	<1200	980
Nitrite	51350	31050	37400
Bromide	<490	<1200	-
Nitrate	161000	111000	118000
Phosphate	3000	<2400	2100
Sulfate	7650	7000	5700
Oxalate	1300	32100	2500
	mmole/mL	**mmole/g**	**mmole/g**
Hydroxide	0.717	-	0.500
	pH	**pH**	
pH	13.32	-	-

2.2 Sr/TRU Removal Conditions

Supernatant from Envelope C waste contains levels of Sr-90 and TRU too high to meet immobilized low-activity waste (ILAW) requirements. The BNFL targets for ILAW are less than 100 nCi/g TRU and less than 20 Ci/m^3 Sr-90 in the final ILAW. For AN-107 waste, this translates to required decontamination factors (DF) of approximately 10 for strontium (90% removal) and 5 for TRU (80% removal). Since over 90% of the TRU in AN-107 is due to Am-241, a decontamination factor of 5 was established for Am-241.

Experimental conditions for Sr/TRU removal were determined from small- and large-scale batch experiments with archived AN-107 waste (Hallen et al. 2000a,b). These experiments suggested that hydroxide could be as low as 0.5M and the reagent addition could be as low as 0.05M strontium and 0.03M permanganate for AN-107. But because the AN-107 diluted feed was more concentrated and contained entrained solids that were not present in the archived AN-107 sample, the conditions used were 1M hydroxide and reagent addition of 0.075M for strontium and 0.05M for permanganate. The target sodium concentration was also reduced to 6.0M, since all previous studies with archived AN-107 had been conducted with more dilute waste. This concentration required an additional dilution/adjustment to the diluted feed with sodium hydroxide solution. The target concentrations for the final treated waste after all chemical additions were set at 6.0M sodium, 1.0M hydroxide, 0.075M strontium, and 0.05M permanganate. The Sr/TRU removal process added additional sodium to the as-received waste giving 79.2 mole or weight percent of the sodium from the original, as-received waste.

2.3 Solids Removal Conditions

A test matrix was developed for conducting crossflow filtration tests using the single element, Cell Unit Filtration (CUF) test stand (Brooks et al. 1999). The test conditions were focused on higher pressure and crossflow velocities of the test matrix for AW-101 based on results from earlier CUF tests. The CUF test conditions are given in Table 2.2. Conditions 9 to 14 could not be tested because the minimum operating volume of the CUF was too large (~700-mL), and the original solids loading (~2%) were too low to reach 20 wt% solids. Approximately 7-L of treated waste would be required to reach the target solids loading of 20 wt% solids.

Table 2.2. Sr/TRU Precipitate Filtration Tests Conditions

Test Number	Sample	Transmembrane pressure (psi)	Crossflow Velocity (ft/s)
0	DI water	10, 20, 30	12.2
1	Feed	50	12.2
2	Feed	30	12.2
3	Feed	70	12.2
4	Feed	Optimum from 2.1 – 2.3	9.1
5	Feed	Optimum from 2.1 – 2.3	15.2
6	Feed	50	12.2
7	Feed	Optimum from 2.1 – 2.7	Optimum from 2.1 – 2.7
8	De-watering	Optimum from 2.1 – 2.7	Optimum from 2.1 – 2.7
9	>20 %wt solids	30	12.2
10	>20 %wt solids	50	12.2
11	>20 %wt solids	70	12.2
12	>20 %wt solids	Optimum from 2.9 – 2.11	9.1
13	>20 %wt solids	Optimum from 2.9 – 2.11	15.2
14	>20 %wt solids	50	12.2
CUF Cleaning			
15	Water/permeate cleaning	N/A	N/A
16	DI water	20	12.2
17	1M nitric acid cleaning (if needed)	N/A	N/A
18	DI water	10, 20, 30	12.2

After the C-104 filtration tests (Brooks et al. 2000) and prior to the Sr/TRU removal tests, the filter element was cleaned by backpulsing and recirculating 1-L of 1M HNO_3 through the CUF. The acid was drained from the CUF and found to be very dark in color, so an additional 1-L of HNO_3 was used to clean the filter element. The second acid wash was considerably cleaner. The CUF system was rinsed until a neutral pH was obtained. The clean water flux of the filter element was determined at 20, 10, and 30 psid (test 0) to evaluate the relative cleanliness of the CUF and filter element. The respective flux rates were 1.38, 0.84, and 1.77 gpm/ft^2 averaged over 20 minutes. These values were 12 to 25% lower than at the start of the C-104 testing. The clean water flux remained relatively constant over the 20 min and the CUF was judged to be clean enough to conduct filtration tests.

After Sr/TRU removal tests, the filter element was cleaned with 1-L of a combination of nitric acid (1M) and citric acid (0.1M). The manganese precipitate, hydrated manganese dioxide, does not dissolve in 1M nitric acid normally used to clean the filter element/CUF system. An alpha-hydroxy carboxylic acid such as citric acid will reduce Mn(IV) to Mn(II), which is soluble in nitric acid. The manganese solids remaining in the CUF and filter element can be dissolved and removed. This chemical treatment, followed by recirculation through an external cartridge filter (Brooks et al. 2000), has been shown to be effective for restoring clean water flux to the filtration unit in earlier tests.

2.4 Experimental

All Sr/TRU and solids removal tests were performed in the High-Level Radiochemical Facility (HLRF) shielded process cells located in the 300 area at Hanford. Approximately 1.8-L of AN-107 diluted feed had been prepared as part of waste characterization. The target composition for the diluted feed was 7.7M sodium and 1.1M hydroxide. The Sr/TRU removal process was demonstrated on 1.4-L of the diluted feed and 0.4-L was saved for future studies. The target values for chemical adjustment and reagent addition are shown in Table 2.3. Stock solutions of the reagents were prepared outside the hot cells for addition to the waste. Sodium hydroxide solution, 3.52M, was added to adjust the sodium and hydroxide concentrations. The strontium solution was made up as the nitrate salt in 1M concentration. The experiment used 1M sodium permanganate. The data show that the final treated waste contained 75 vol% of the diluted feed or 79.2 wt% of the original, as-received waste sodium.

Table 2.3. Chemical Additions to AN-107 Diluted Feed

	Target Concentration (M)	Actual Concentration (M)	Density (g/mL)	Target Volume (L)	Actual Volume (L)	Sodium from As-Received Waste (wt %)
initial waste	-	-	1.32	1.40	1.398	86.4
NaOH	3.52	3.51	1.123	0.265	0.264	-
$Sr(NO_3)_2$	1.00	0.999	1.159	0.143	0.143	-
$NaMnO_4$	1.00	1.001	1.094	0.097	0.096	-
final waste	-	-	1.298	1.90	1.86	79.2

AN-107 diluted feed was transferred from four, 1-pint storage jars to a 4-L Erlenmeyer flask. The waste was mixed with a magnetic stir bar while reagents were slowly added in the order shown in Table 2.3. The waste was thoroughly mixed between each reagent addition. After the permanganate addition, the waste was mixed for 30 min at ambient temperature, and then heated for 4 hours at 50°C. The treated waste was cooled and transferred to the CUF feed reservoir for filtration tests. The test matrix was run; the slurry diluted by approximately 20%, and CUF tests repeated at a lower slurry concentration to determine the impact on filtration performance. The dilution did not add additional sodium to the waste so 79.2 mole or weight percent of the sodium in the final treated waste was from the original, as-received waste.

2.5 Sampling Plan

A sampling plan (Table 2.4) was prepared before the Sr/TRU removal test was performed. The sampling plan was based on the Test Specification, comments received from external reviewers (need for duplicates), and past experience (collect additional samples during tests). Not all of the samples were analyzed. The samples that were not needed for analytical were recombined at the end of the test with the CUF slurry to maximize the amount of supernatant for downstream processing. Chemical analyses also included the radioactive elements.

Table 2.4. Sampling Plan

Sampling Step	Number of Samples	Sample Type	Process Step	Analysis
Precipitated Feed	2	Slurry	After Precipitation	Chemical Analyses
Digested Precipitate	2	Slurry	After Digestion	Chemical Analyses
Recycled Slurry	2	Slurry	CUF slurry sample	Physical Properties
Recycled Slurry	2	Slurry	CUF Slurry Sample	Rheology
CUF Test Matrix	4	Filtrate	CUF Filtrate sample	Chemical Analyses
Middle De-water Step	1	Filtrate	During Condition 8	Chemical Analyses
Diluted CUF Slurry	1	Slurry	Diluted CUF slurry sample	Physical Properties
Diluted CUF Slurry	1	Filtrate	Diluted CUF Filtrate	Chemical Analyses
Filtrate Composite	2	Filtrate	Combined CUF & Deadend Filtrates	Chemical Analyses
Four Washes	1 each	Wash	After Each Wash	Chemical Analyses
Wash Composite	1	Wash	After All Washes	Chemical Analyses
Washed Solids	2	Wet Solids	Final Solids	Chemical Analyses

2.6 Chemical Analyses

All of the chemical analyses were conducted at Battelle. BNFL designated the analytes of interest and minimum reportable quantity in the test specification (Table 2.5). Table 2.6 lists the samples that were analyzed and analyses conducted on each sample. The samples were transferred from the HLRF to the Shielded Analytical Laboratory (SAL) for sample preparation and analyses. The samples were analyzed as soon as possible, but for some samples this was up to two weeks later. The data discussed in Section 3.0 shows that the samples that were taken as slurries, continued to react during sample storage.

Table 2.5. Analytical Requirements for Washed Solids, Filtrate, and Wash Solutions

Analyte	**Washed Solids Minimum Reportable Quantity (MRQ) µCi/g**	**Filtrate and Wash Solutions Minimum Reportable Quantity (MRQ) µCi/mL**
Cesium-137	6.0E-02	9.0E+00
Strontium-90	7.01E+01	1.5E-01
Technetium-99	6E+00 µg/g	1.5E-03
Americium-241	1.2E-03	7.2E-04
Europium-154	6.0E-02	2.0E-03
Europium-155	6.0E-02	9.0E-02
Plutonium-239/240	6.0E+00 µg/g	9.6E-03
Total Alpha	1.0E-03	2.3E-01
	µg/g	**µg/mL**
Al	3.3E+02	7.5E+01
Ba	6.0E+02	7.8E+01
Ca	1.8E+02	1.5E+02
Cd	1.1E+01	7.5E+00
Co	3.0E+00	3.0E+01
Cr	1.2E+02	1.5E+01
Cu	1.8E+01	1.7E+01
Fe	1.4E+02	1.5E+02
K	1.5E+03	7.5E+01
La	6.0E+01	3.5E+01
Mg	5.4E+02	1.5E+02
Mn	3.0E+02	1.5E+02
Mo	6.0E+00	9.0E+01
Na	1.5E+02	7.5E+01
Ni	1.6E+02	3.0E+01
Pb	6.0E+02	3.0E+02
Si	3.0E+03	1.7E+02
Sr	3.0E+02	8.7E+01
Ti	1.5E+02	1.7E+01
U	6.0E+02	6.0E+02
Zn	6.0E+00	1.65E+01
TOC	6.0E+01	1.5E+03
TIC	3.0E+01	1.5E+02
Cl	2.3E+02	3.0E+00
F	7.5E+03	1.5E+02
NO3	4.5E+02	3.0E+03
SO4	1.2E+03 (as S)	2.3E+03
PO4	6.0E+02 (as P)	2.5E+03

Table 2.6. Identification of Samples that were Analyzed[1]

Sample Number	Type	Comments
DF-01	filtrate	Analyze for ^{90}Sr, ^{241}Am, and Na (and all ICP elements)
DF-02	slurry	filter and analyze for ^{90}Sr, ^{241}Am, and Na
DF-03	slurry	filter and analyze for ^{90}Sr, ^{241}Am, and Na
DF-04	slurry	filter and analyze for ^{90}Sr, ^{241}Am, and Na
DF-05	slurry	physical properties
DF-06	slurry	physical properties
DF-11	CUF filtrate	analyze for ^{90}Sr, ^{241}Am, and Na
DF-13	dilute slurry	physical properties
DF-14	dilute slurry	filter and analyze for ^{90}Sr, ^{241}Am, and Na
DF-20	composite filtrate	analyze for all analytes, see Table 2.5
DF-21	composite filtrate	analyze for all analytes
1st wash	wash	analyze for Na (and all ICP elements)
2nd wash	wash	analyze for Na
3rd wash	wash	analyze for Na
4th wash	wash	analyze for Na
wash composite	combined washes	analyze for all analytes
washed solids	damp solids	Dry, digest, analyze for all analytes

[1]See Table 3.1 for description of process conditions corresponding to each sample.

Sample DF-01 was filtered immediately after the sample was collected. The other slurry samples, when specified, were filtered as part of the analytical sample preparation scheme, about 2 weeks after the samples were taken. The slurry samples were filtered with 0.45-μm disposable syringe filters. Many of the samples required multiple filter units to filter the necessary volume of waste for analyses. The washed solids were dried for 24 hours at 105°C, and the weight percent of dry solids was determined. Duplicate samples of the dried solids were dissolved by acid-digestion and analyzed for Na/K (and all ICP elements). Duplicate, washed solids samples were also dissolved by potassium hydroxide-fusion and analyzed for all analytes listed in Table 2.5. The wash samples were analyzed separately for sodium (and all ICP elements), and a composite of all four washes made; 5 mL of each wash combined to make one 20-mL sample. The composite wash was analyzed for all analytes (Table 2.5).

2.7 Physical Property Measurements

Physical property measurements were conducted on samples of AN-107 diluted feed before and after treatment. The physical property analyses included supernatant and centrifuged solids density, and weight percent (wt%) and volume percent (vol%) solids.

An AN-107 diluted feed sample (sample identification "AN-107 PT") was collected from the initial diluted tank composite. The diluted feed was homogenized and sampled as described under Test Plan 29953-6. This sampling method was shown under Test Plan 29953-1 to provide samples with representative solids content.

The AN-107 diluted feed was treated for Sr/TRU removal under Test Plan 29953-013 according to Test Instruction 29953-052. Duplicate samples of the Sr/TRU precipitated slurry (DF-05 and DF-06) were collected for physical property analyses. The Sr/TRU precipitated slurry was then diluted by 19.24% for additional filtration tests, and an additional slurry sample (DF-13) was collected for physical property analysis.

2.8 Rheological Properties Testing

The AN-107 initial diluted feed (AN-107 PT) and Sr/TRU precipitated slurry (similar to DF-05 and DF-06) were analyzed for shear stress as a function of shear rate from approximately 0.1 to 300 s^{-1} or 1000 s^{-1} according to procedure 29953-010. The AN-107 initial diluted feed was analyzed using the Bohlin CS viscometer modified for glovebox operations. Concentric cylinders with a 25-mm-diameter inner cylinder and a "Small Sample Cell" outer cylinder were used as the measuring geometries. The Sr/TRU precipitated slurry was analyzed using a Haake M5 measuring head modified for hot cell operations. An MVI measuring geometry was used on the Haake. Both the initial and Sr/TRU precipitated slurries were analyzed in duplicate at 25°C. A 49.9 cP standard, Brookfield lot 102298, was used to check the calibration of both instruments before samples were analyzed. The Bohlin is the preferred instrument for analyzing the diluted feed as it can quantify lower viscosities than the Haake M5 with comparable concentric cylinder geometries. The Bohlin also requires significantly less sample material, 5 ml, compared to 40 ml for the Haake M5. Since the Sr/TRU slurries were analyzed in the hot cell while the filtration tests were being conducted, the Haake M5 was preferred for these analyses. The Haake M5's location in the hot cell allowed for immediate return of sample to the CUF reservoir following analysis, preventing the loss of sample volume from the test.

Analyses of the AN-107 initial diluted feed were conducted up to 300 s^{-1} as per Test Instruction 29953-11. Following this testing, guidance was provided by BNFL to increase the shear rate analyses to 1000 s^{-1}. Therefore, testing on the Sr/TRU precipitated slurries were conducted up to 1000 s^{-1} with additional analyses conducted to only 300 s^{-1} for comparison.

Prior to shear stress as a function of shear rate analysis, the samples were stirred to combine the separated liquid and solid layers. Shear stress data, as a function of shear rate, was obtained by measuring the shear stress produced at a specific shear rate. The shear rate was gradually increased from approximately 0.1 to either 300 s^{-1} or 1000 s^{-1}, generating the increasing shear rate curve, and then back down to 0.1 s^{-1}, generating the decreasing curve. For analyses conducted with the Haake M5, the increasing and decreasing 300 s^{-1} curves were collected over a 2 min period, while the 1000 s^{-1} curves were collected over a 2- or 4-min time period. For the Sr/TRU precipitated slurry, the shear rate analysis was conducted three times with the same sample still in the instrument. A difference between the first, second, and third analysis of the same sample would indicate potentially unusual behavior in the samples including (but not limited to) settling of the solids within the instrument, the sample being affected by shearing in the instrument or water loss through evaporation. In all cases, the first, second, and third analyses were virtually identical. The sample cup was then cleaned, and a duplicate sample was analyzed using the same parameters.

3.0 RESULTS AND DISCUSSION

The results of the testing and analyses are discussed below for Sr/TRU decontamination, chemical composition, wash solutions and washed solids composition, solids removal, physical properties, and rheological and flow properties.

3.1 Sr/TRU Decontamination

Multiple samples were taken during the Sr/TRU removal tests and analyzed to determine the change in waste composition upon treatment. Samples were taken after various stages of treatment, filtration, and again after the slurry was further diluted for additional filtration tests. The radionuclide composition of the treated samples was compared with the starting composition to determine the extent of decontamination. The Decontamination Factor (DF) is defined as the concentration of the component in the untreated waste divided by the concentration after treatment, corrected by the amount of dilution that occurred:

$$DF = [A]_i/([A]*MD)$$

where $[A]_i$ is the concentration of component A per mass in the diluted feed sample, [A] is the concentration of component A per mass in the treated sample, and MD is the mass dilution, final mass of treated solution divide by the initial mass of solution. The final mass is determined by summing up the mass of initial waste and all dilution, adjustments, and/or reagent additions. Table 3.1 lists the samples analyzed, mass dilution to be used for calculating DFs, and description of the sample. All DFs and mass dilutions are based on the diluted feed.

Table 3.1. Samples and Mass Dilution for Calculating Decontamination Factors

Sample ID	Mass Dilution	Sample Description
DF-01	1.3075	sampled after chemical addition, before heating, and filtered immediately
DF-02	1.3075	same as DF-01 but left as slurry until sample prep.
DF-03	1.3075	sampled after heating, left as slurry until sample prep
DF-04	1.3075	duplicate of DF-03
DF-11	1.3075	CUF filtrate
DF-14	1.556	diluted CUF slurry, left as slurry until sample prep
DF-20	1.556	composite filtrate from CUF and Deadend filtration, also Cs ion exchange feed
DF-21	1.556	duplicate of DF-20, composite filtrate

The strontium, americium, and curium decontamination factors for samples DF-01 through DF-11 are shown in Table 3.2. All samples had very high decontamination for Sr and TRU components, greatly exceeding the requirements for ILAW. The 4-hour digestion had very little effect on the DFs. The higher DFs for samples DF-02, DF-03, and DF-04 most likely resulted from the long contact time of the solids and supernatant (approximately 2 weeks), while the samples were waiting preparation in the analytical laboratory. This suggests that thermodynamic equilibrium was not reached between solution species and solids during the actual waste testing. Sample DF-11 most accurately represents the current technical baseline process for Sr/TRU removal because it underwent the entire treatment and filtration process.

Table 3.2. Strontium, Am, and Cm Decontamination Factors for Samples DF-01 to DF-11 and the Composition of CUF Filtrate (DF-11)

	DF-01	**DF-02**	**DF-03**	**DF-04**	**DF-11**	**DF-11 (μCi/g)**
Sr-90	48	116	246	122	82	5.85E-01
Am-241 (AEA)	26	54	64	56	28	9.36E-03
Cm-242	24	39	49	42	22	3.89E-04
Cm-243+Cm-244	18	37	43	39	20	4.17E-05

The original treated waste was diluted by an additional 19 wt%, additional CUF tests performed, and samples collected for analyses. A reduced test matrix was performed and the optimum conditions were used to dewater the slurry in the CUF until the waste volume was reduced to the volume limit of the CUF. Approximately half the slurry was dewatered in the CUF. The CUF was drained and the slurry filtered to dryness in a deadend filtration unit. The final CUF filtrate was combined with filtrate from the final dewatering of the slurry in a deadend filtration unit and sampled in duplicate, DF-20 and DF-21. These samples represent the low activity waste from the Sr/TRU removal process. The composite filtrate is the feed for the subsequent cesium ion exchange process.

Table 3.3 shows the DFs calculated for the total sample (including entrained solids) and for supernatant for various radioactive elements in these samples. Solids removal contributed very little to the DF. The DFs were very high and consistent with the earlier filtrate sample, DF-11. The DF of less-than-1 for Tc-99 means the concentration after treatment was actually higher in the filtrate than the untreated waste. The most significant result was the additional dilution did not impact the decontamination of the waste.

Table 3.3. Radioactive Element Decontamination Factors for Samples DF-14, DF-20 and DF-21 and Average Composition of Composite Filtrate

	Total Sample			Supernatant Only			Average of DF-20 & DF-21 (µCi/g)
	DF-14	DF-20	DF-21	DF-14	DF-20	DF-21	
Co-60 (GEA)	-	1	1	-	1	1	4.99E-02
Sr-90	55	85	84	51	78	77	4.77E-01
Cs-137 (GEA)	-	1	1	-	1	1	1.23E+02
Eu-154 (GEA)	-	11	12	-	10	11	2.85E-02
Pu-238	-	29	29	-	22	23	1.67E-04
Pu-239+Pu-240	-	29	31	-	24	25	6.32E-04
Am-241 (AEA)	66	45	43	56	38	37	4.97E-03
Cm-242	51	35	32	46	32	29	2.32E-05
Cm-243+Cm-244	50	30	27	46	27	25	2.32E-04
Tc-99	-	0.8	0.7	-	-	-	2.72 (µg/g)
Total Alpha	-	32	34	-	27	28	7.93E-03

3.2 Change in Chemical Composition

The Sr/TRU removal process also changed the chemical composition of the waste samples. Table 3.4 shows the compositional change of the supernatant as a percent removed from the untreated waste. These values are corrected by the mass dilution that occurred upon treatment. The most significant and interesting changes are for Cr, Fe, Mn, and Sr. The Cr in solution decreased, which was opposite of the effect observed by Orth et al. The Fe removal was very high, and most likely correlated directly with the high TRU (Am/Cm/Eu) removal. Manganese decreased, which suggests that soluble Mn, likely Mn(II), is oxidized to Mn(IV) by reaction with Mn(VII). The Sr increased because the original solution was below the saturation level for Sr, and a fraction of the added Sr remained soluble.

Table 3.4. Percent Removal of ICP Metals from samples DF-01 to DF-11 and Composition of the CUF Filtrate (DF-11)

	DF-01 (%)	DF-02 (%)	DF-03 (%)	DF-04 (%)	DF-11 (%)	DF-11 (µg/g)
Al	10	7	7	5	7	2220
Ca	32	36	39	38	37	157
Cd	0	-4	-3	-5	-3	27.1
Cr	48	93	94	93	51	51.5
Fe	99	99	99	99	98	15.9
K	24	23	24	23	23	732
Mn	99	94	92	93	99	1.8
Ni	2	-1	-1	-3	-1	222
P	0	-4	-3	-5	-4	294
Pb	65	65	65	64	60	65.9
Sr	-7445	-4132	-4113	-4216	-7070	153
Zr	52	72	75	72	57	3.4

The results for the diluted slurry and final composite filtrate are presented in Table 3.5. The results show the composition did not change significantly upon dilution of the slurry and additional CUF testing.

Table 3.5. Composition Change, Reported as Percent Removed, on Treatment and Dilution of the Slurry and Final Composite Filtrate

	DF-14 (%)	DF-20 (%)	DF-21 (%)	Ave DF-20 & 21 (µg/g)
Al	5	-1	3	1980
Ca	33	32	37	136.5
Cd	-3	-7	-2	23.2
Cr	85	58	59	37.025
Fe	99	99	99	8.9125
K	24	21	23	622.5
Mn	98	99	99	2.325
Nd	80	74	74	2.725
Ni	-2	-7	-1	192
P	-4	-8	-4	251.5
Pb	61	60	62	54.675
Sr	-5164	-6341	-5867	111.25
Zr	77	68	70	2.05

Table 3.6 shows the results from sodium analyses of the samples and the density of the filtrate. The sodium concentration is important for LAW glass-loading calculations. Here, the sodium values are higher than expected, 6M for the initial treated waste and 5M after additional dilution. These values could be caused by dissolution of additional entrained solids on dilution/treatment, but the results are within the expected analytical error (10%) for samples that require high dilution before analysis. The densities reported in the table were determined in the hot-cells using volumetric flasks and samples of the filtrate.

Table 3.6. Sodium Concentration in DF-01 to DF-21 Samples

Sample ID	[Na] (µg/g)	[Na] (M)	Density (g/mL)
DF-1	112000	6.27	
DF-2	115000	6.43	
DF-3	116000	6.49	
DF-4	118000	6.60	
DF-11	119000	6.66	
average	116000	6.49	1.286
DF-14	105000	5.67	
DF-20	102000	5.51	
DF-20 dup.	110000	5.94	
DF-21	91900	4.96	
average	101300	5.47	1.2414

Carbon analyses were only performed on the composite filtrate, samples DF-20 and 21. Table 3.7 shows that very little of the organic carbon was destroyed by the Sr/TRU removal process, i.e. permanganate oxidation. This is consistent with the initial high organic carbon content of the waste (>2M) and low level of added permanganate (0.05M). The decrease in carbonate is expected because the added Sr precipitates as $SrCO_3$, removing carbonate from solution. Taking into account the carbonate removed by Sr precipitation, the carbonate (reported as TIC) still decreased by 6%. The data indicate very little destruction of organic carbon and give no indication that any was oxidized to carbonate.

Table 3.7. Percent Removal of Carbon and Composition of the Composite Filtrate Samples

	DF-20 (%)	**DF-21 (%)**	**Average (µg/g)**	**Average (M)**
TIC	14	13	6800	0.70
TOC	3	9	13600	1.41
TC	7	10	20425	2.11

The filtrate composite, DF-20 and DF-21, was analyzed by ion chromatography (IC) for anions. Table 3.8 shows that most anions were below detection level, except for nitrate and nitrite. The Sr/TRU removal process reduced the nitrite concentration about the same amount as the nitrate was increased, which suggests that during the removal process some of the nitrite was oxidized to nitrate. Not enough data are available to determine if this is a result of the initial permanganate oxidation, or a result of the digestion process, where various higher valence metals may act as nitrite oxidants (Mn(IV)).

Table 3.8. Percent Removal of Anions and Composition of the Composite Filtrate Samples

	DF-20 (%)	**DF-21 (%)**	**Average (µg/g)**	**Average (M)**
Fluoride	>33	>33	<2000	
Chloride			<2000	
Nitrite	9	10	21700	0.59
Nitrate	-6	-8	87400	1.75
Phosphate			<4000	
Sulfate			<4000	
Oxalate			<4000	

3.3 Composition of Wash Solutions and Washed Solids

The combined entrained solids and Sr/TRU removal solids were collected on the deadend filter. The solids were washed on the filter four times with 0.01M NaOH. The first wash was vacuum filtered, but filtered so fast the solids were not washed very well. Subsequent washes were all done with gravity filtration. The solids were very difficult to wash in the deadend filtration unit. They could not be easily stirred or mixed while washing. The solids were the consistency of wet clay. The four wash solutions were collected and analyzed separately, and a composite of the four wash solutions was analyzed. The wet, washed solids were removed from the filter unit, dried to constant weight, dissolved, and analyzed. The composition of the wash solutions and washed solids are listed in Table 3.9.

Table 3.9. Composition of Wash Solutions and Washed Solids (µg/g)

Element	1st Wash*	2nd Wash	3rd Wash	4th Wash	Composite Wash	Washed Solids
Ag	-	-	-	-	-	105
Al	194	487	161	90.8	125.9	7915
Ba	-	-	-	-	-	358
Ca	15.7	[42]	16.6	[10]	13.3	5413
Cd	1.9	5.29	1.8	1.07	1.435	33
Ce	-	-	-	-	-	1013
Co	-	-	-	-	-	-
Cr	1.19	[2.8]	2.52	1.77	2.145	3277
Cu	1.38	[3.3]	[1.1]	[0.62]	0.86	53
Fe	[1.1]	[1.4]	3.44	[0.44]	1.94	47133
K	[73]	[140]	[34]	[14]	24	-
La	-	-	-	-	-	723
Mg	-	-	-	-	-	195
Mn	[0.52]	-	[0.52]	[0.28]	[0.4]	130000
Mo	[1.2]	[3.1]	[1]	[0.56]	[0.78]	31
Na	12400	26500	9560	6440	8000	75567
Nd	-	-	-	-	-	2117
Ni	16.2	43.6	14.8	8.88	11.84	134
P	18	61.4	11.7	11.3	11.5	749
Pb	9.14	23.9	9.82	7.03	8.425	4770
Pd	-	-	-	-	-	987
Si	27.2	[18]	[18]	[10]	14	4322
Sr	16.7	40	16.7	12.4	14.55	272500
Th	-	-	-	-	-	1150
Ti	-	-	-	-	-	42
U	-	-	-	-	-	1780
Zn	0.69	1.8	0.8	0.6	0.7	307
Zr	-	-	-	-	-	1958

* First wash vacuum filtered, all others gravity filtered. Overall error is estimated to be within ±15%. Values in brackets are within 10-times the detection limit and errors are likely to exceed ±15%.

Table 3.9. Composition of Wash Solutions and Washed Solids (Con't)

	Wash Composite (µCi/g)	**Washed Solids (µCi/g)**
Alpha	6.67E-04	1.35E+01
Sr-90	9.79E-02	1.10E+03
Pu-239 + Pu-240	2.44E-05	6.33E-01
Pu-238	6.97E-06	1.76E-01
Am-241	2.89E-04	6.24E+00
Cm-243 + Cm-244	1.62E-05	1.40E-01
Cm-242	1.39E-06	1.07E-02
	(µg /g)	**(µg /g)**
Tc-99	0.29	8.6
TIC	1,025	47,750
TOC	2,400	1,625
TC	3,425	49,375
Fluoride	<125	<250
Chloride	<125	<500
Nitrite	2800	1750
Nitrate	11150	8350
Phosphate	<250	<500
Sulfate	470	1200
Oxalate	3450	23300

The moisture content of the washed solids (92 g) was 63.4 wt%, which yielded 33.7 g of solids recovered on a dry basis. The Sr/TRU removal treatment was estimated to yield 35 g of solids, and the initial waste was estimated to contain 11 g of entrained solids. The loss of solids occurred because complete removal of the dewatered slurry from the CUF is difficult. Additional entrained solids were also expected to dissolve by the additional dilution/treatment, lowering the amount of entrained solids. Using the Sr concentration of the washed solids (272,500 µg/g) and predicted Sr concentration for the solids from precipitation (345,000 µg/g), we calculate that 9 g of entrained solids were present. The solids content of the waste slurry is calculated to be 1.85 wt% and includes the entrained solids and Sr/TRU precipitate. The calculation suggests the entrained solids are primarily Na, Al, Si, Fe and Mn, which is consistent with data on entrained solids washing/dissolution (Lumetta et al. 1999). The IC analysis was performed on a water leach of the solids and TIC/TOC analyses were made directly on the solids. However, the TOC and oxalate results reported in Table 3.10 for the washed solids did not match. With oxalate at 23,300 µg/g, the TOC value should have been at least 6350 µg/g. It is likely that the oxalate decomposed during the TIC analyses and is reported as TIC not TOC.

Table 3.10. Average Filtrate Flux as a Function of Test Number and Conditions

Test #	TMP (psi)	Total Flow (gpm)	Velocity (ft/s)	Average Flux (gpm/ft^2)
1	49	4.0	12.0	0.030
2	30	4.2	12.2	0.025
3	69	3.4	9.9	0.022
4	50	3.1	9.0	0.019
5	49	4.5	13.1	0.024
6	50	4.1	11.9	0.022
DW	48	3.9	11.3	0.021

3.4 Solids Removal

The CUF was thoroughly cleaned before each test matrix of filtration tests. The flux rate for clean water was used to evaluate the cleanliness of the CUF unit. When the flux rate was high, and remained consistently high for at least 30 min, the CUF was judged clean enough for filtration tests. The Sr/TRU precipitated waste was then transferred to the CUF for testing.

The result from the first test condition, target transmembrane pressure (TMP) of 50 psi and crossflow velocity of 12.2ft/s, are shown in Figure 3.1, and are indicative of the overall CUF performance for the Sr/TRU precipitated waste. The high initial flux rate drops within a few minutes to a lower, consistent flux rate that slowly decreases over the 1-hour test period. For comparison of test conditions, the flux rate is averaged over the 1-hour run time except for the initial flux rate data (data taken at less than 10 min). Averaging the data from 10 min until the end of the test conditions, the average flux rate was 0.03 gpm/ft^2 or 1.75 m^3/m^2/day and the permeability was 0.53 m/day/bar. These flux rates are significantly less than the value of 0.1 gpm/ft^2 that was obtained for the Sr/TRU precipitated, archived AN-107 under similar conditions. The archived waste did not have entrained solids present and was more dilute. The filter element also had a slightly higher clean water flux, which suggests the filter element and CUF was initially cleaner for the archived AN-107 sample test.

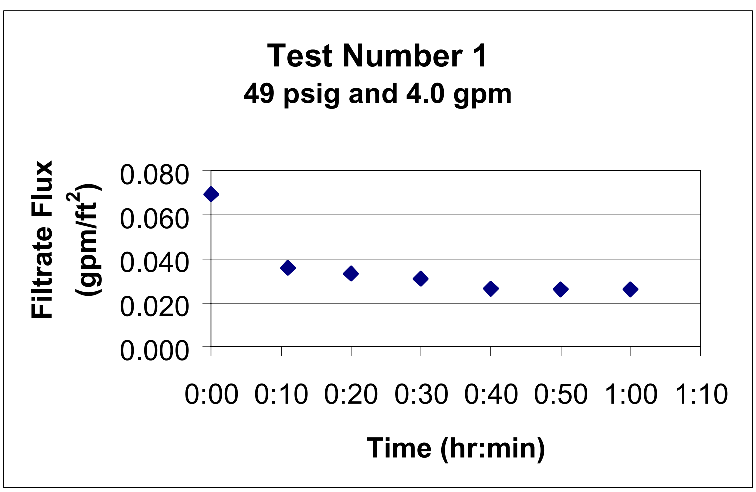


Figure 3.1. CUF Data for Test Number 1 of the Test Matrix, 49 psi Transmembrane Pressure and 4.0 gpm Total Flow (crossflow velocity of 12 ft/s)

Comparing all of the data from the test conditions, Table 3.10, the important operating factor appears to be crossflow velocity. The flux rates appear to drop fairly consistently with increasing run number, which has been observed for other wastes. However, for test condition number 5, the flux rate increases substantially over the previous two conditions. The decrease in flux rates with run number, or time, can be seen by comparing the results for test number 1 and number 6 (Figure 3.2). The average filtrate flux had decreased from the first test, 0.030 gpm/ft^2, to 0.022 gpm/ft^2 for test condition 6. If the decreases in flux rates are estimated for each test, the effect of TMP and velocity can be determined.

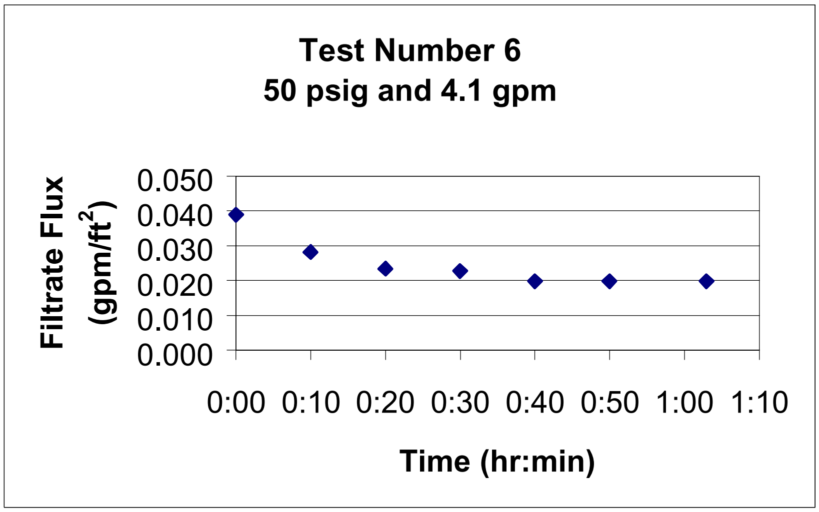


Figure 3.2. CUF Data for Test Number 6, Repeat of Test Number 1

Increasing the TMP from 30 to 50 psi resulted in a 13% increase in filtrate flux, but increasing the TMP another 20 psi from 50 to 70 psi, resulted in only a 2% increase in filtrate flux. Similarly, increasing crossflow velocity from 9.0 to 11.6 ft/s resulted in a 31% increase in flux, but an additional increase from 11.6 to 13.1 ft/s resulted in only a 2.5% increase in flux. Unfortunately, because of pump wear, the high pressure and crossflow velocity test conditions could not be reached, 70 psi and 12.2 ft/s, and 50 psi and 15.2 ft/s. However, the results that were obtained suggest these conditions would not have significantly increased filtrate flux compared with 50 psi and 12.2 ft/s.

The conditions for test 1 and 6 were chosen as optimum and used for dewatering the waste in the CUF. The target TMP of 50 psi and total flow of 4.1 gpm could not be sustained during dewatering because of the decreasing slurry volume with time. The waste volume was decreased more than 50% during dewatering, and the filtrate flux remained quite constant during dewatering, as shown in Figure 3.3. Dewatering was stopped at 38 min because the volume in the CUF was too low to continue. The average flux rate during dewatering was 0.021 gpm/ft^2.

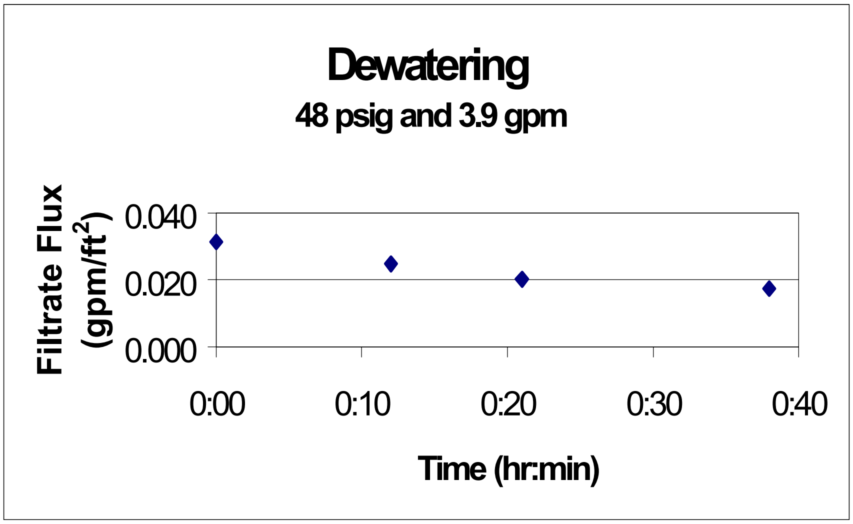


Figure 3.3. Flux Rate During Dewatering in the CUF Unit

The CUF testing was completed and the remaining dewatered slurry removed from the CUF. The CUF was rinsed with DI water and cleaning of the system was initiated. Preliminary evaluation of the filtrate flux data for the Sr/TRU treated AN-107 diluted feed showed a significantly lower filtrate flux than for the treated archived AN-107 sample. Figure 3.4 shows a comparison of the first three test conditions for the treated archived AN-107 sample and the treated AN-107 diluted feed. The large drop in filtrate flux between condition 1 and 2 for the treated, archived AN-107 was thought to be a result of shearing the agglomerated precipitate (solids) into smaller particles. This behavior was not noticed for the treated AN-107 diluted feed.

Additional CUF tests were performed to determine if waste concentration could be a major factor in filter performance. The filtrates, slurry/solids, and samples from the first series of CUF tests were combined to reconstitute the Sr/TRU removal slurry, ~2 wt% solids. This waste was transferred back to the CUF and filter performance determined using test condition 1. Then the waste slurry was diluted by 19 wt% with DI water from the first CUF rinse (~1.7 wt% solids), and filtration tests repeated: test conditions 1, 2, 4, 6, and dewatering, plus an additional test to assess the impact of frequent backpulsing every 10 min of operation (test #7). Figure 3.5 shows the filtrate flux rates for the starting reconstituted waste and the waste after additional dilution, for similar test conditions, test condition 1. The reconstituted waste average flux rate was 0.020 gpm/ft^2. After dilution it increased to 0.028 gpm/ft^2, a 36% improvement. The dilution of the waste improved the flux rate, but not to the high values obtained for filtration of the treated archived AN-107.

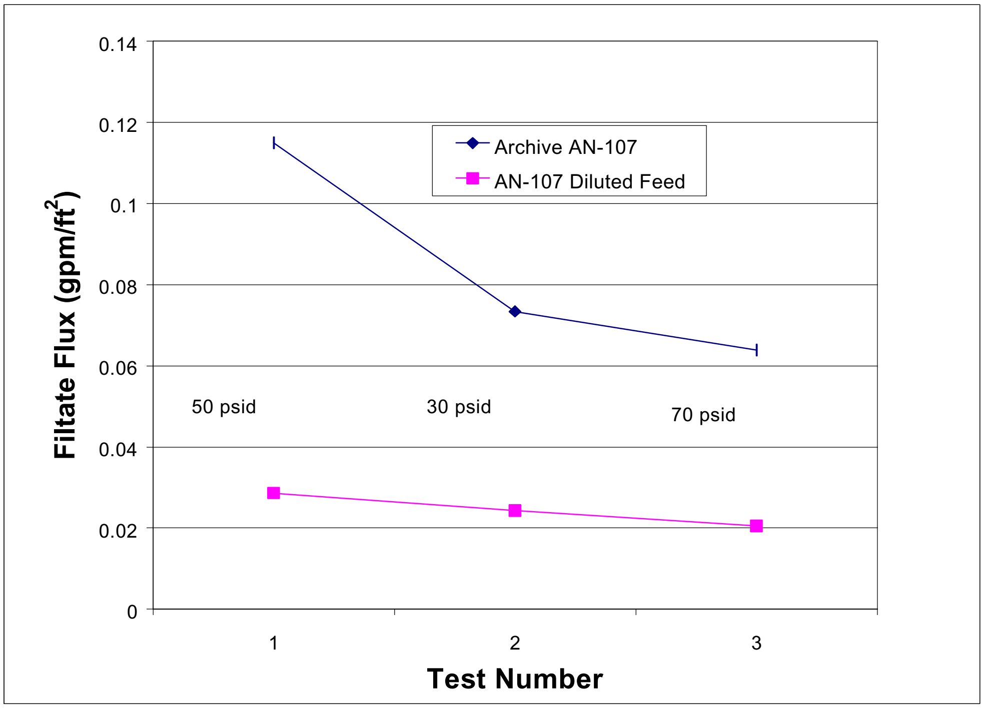


Figure 3.4. Comparison of Flux Data from Sr/TRU Precipitated Archived AN-107 and AN-107 Diluted Feed

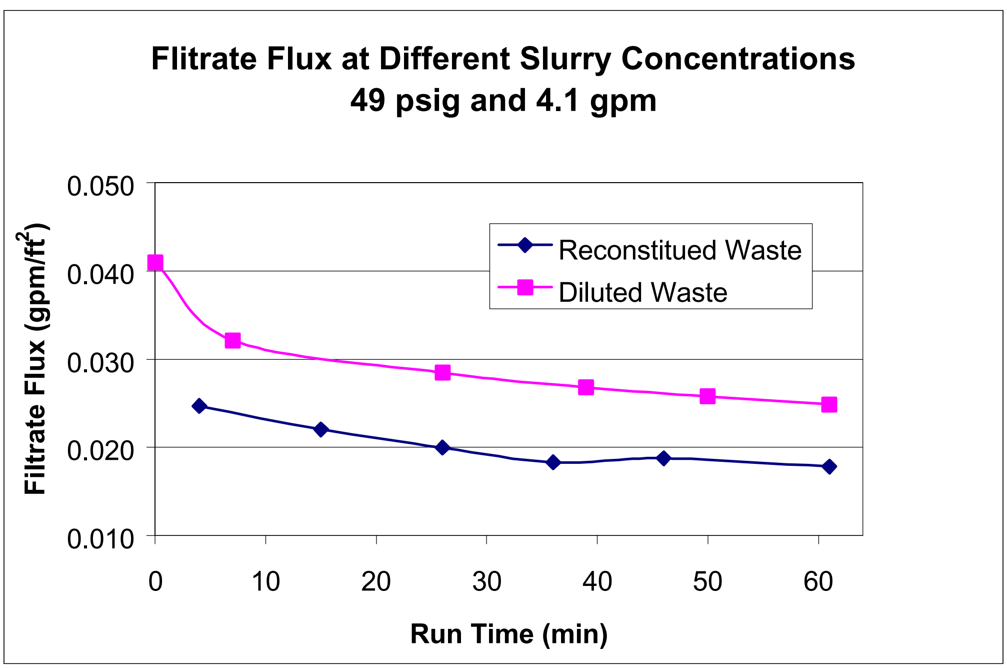


Figure 3.5. Comparison of Flux Rates at Different Slurry Concentrations with Test Number 1 Conditions

Table 3.11 gives the filtrate flux rates for the diluted slurry at various conditions. The data are consistent with the tests conducted with the more concentrated slurry. Increasing pressure from 28 to 49 psi resulted in a 14% increase in flux. Increasing crossflow velocity from 9.0 to 11.9 ft/s resulted in a 27% increase in flux. The filtrate flux decreased as run number increased, but to a lesser extent than with the more concentrated slurry. Optimum conditions for dewatering were determined to be 50 psi and 12 ft/s as with the more concentrated slurry. Dewatering the diluted slurry gave very similar results to the more concentrated slurry.

Table 3.11. CUF Test Results for the Diluted Slurry

Test #	TMP (psi)	Total Flow (gpm)	Velocity (ft/s)	Average Flux (gpm/ft^2)
1	49	4.1	11.9	0.028
2	28	4.2	12.2	0.024
4	49	3.1	9.0	0.021
6	49	4.1	11.9	0.026
7	49	4.1	11.9	0.029
DW	48	3.9	11.3	0.022

The average flux value reported for test condition 7, the frequent backpulse test, excludes the initial flux rate measurements. If the initial flux values are included in the average, the value raises slightly to 0.032 gpm/ft^2. The backpulse procedure is described in Brooks et al. (1999). The air pressure to the backpulse chamber was set to 65 psig. A backpulse would take approximately 8 seconds to complete. The number of backpulses was varied from 1 to 2. The filtrate flux rates and number of backpulses used are shown in Figure 3.6 as a function of filtration time. The initial, time 0, flux rates should not be used for comparison, because slight delays between back pulse and taking the reading can cause the values to be significantly different as the initial rate is changing rapidly. Comparing the flux rate data taken at approximately 10 min, shows continued degradation of flux rates even with frequent backpulses. Going from 1 backpulse to 2 appeared to stop or temporarily delay the degradation of filter performance, but did not recover lost performance. The significant factor affecting the flux rates with regard to the earlier archived AN-107 data appears to be an overall reduction of filter element/CUF performance with time. The entrained solids present in the AN-107 diluted feed certainly had an effect but probably similar in magnitude to the impact of dilution.

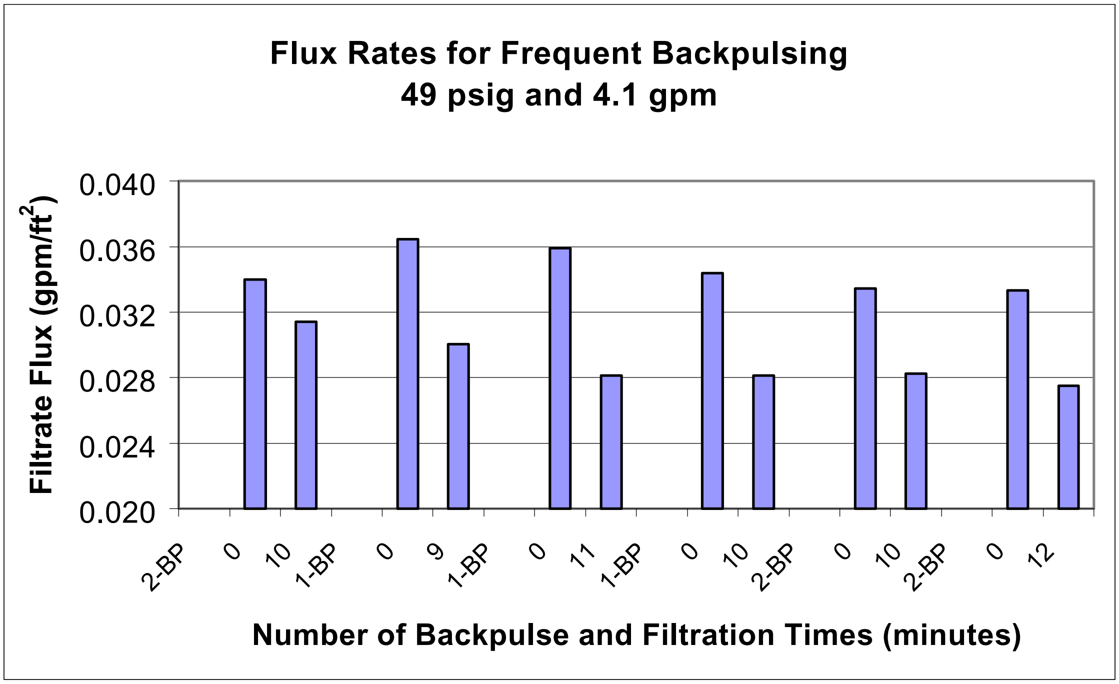


Figure 3.6. Filtrate Flux Rates with Frequent Backpulsing

3.5 Physical Properties Analysis

The AN-107 samples (AN-107 Diluted Feed (AN-107 PT), Sr/TRU Precipitated Slurry (DF-05 and DF-06), and Diluted Sr/TRU Precipitated Slurry (DF-13)) were analyzed for density of the bulk slurries, settled solids, supernatant following solids settling, centrifuged solids, and centrifuged supernatant. The density results are listed in Table 3.12. The wt% and vol% settled solids, wt% and vol% centrifuged solids, and wt% total solids were measured for these samples as well. The wt% and vol% solids results are listed in Table 3.13.

For this testing, a known mass of each slurry was placed in duplicate in volume graduated centrifuge cones. The duplicates were then allowed to settle for 3 days. The total mass (M_B) and volume (V_B) of the settled were recorded, and the density of the bulk slurry was calculated ($D_B=M_B/V_B$). In addition, the volume of the settled solids (V_{ss}) and volume of supernatant following solids settling (V_{sl}) were recorded. The vol% settled solids were then calculated ($Vol\%_{ss}=V_{ss}/V_B$ x 100%). A portion of the supernatant following solids settling was then transferred to a graduated cylinder, and its mass (M_{slb}) and volume (V_{slb}) recorded. Using these data, the density of the supernatant following solids settling was calculated ($D_{sl}=M_{slb}/V_{slb}$).

Since all of the supernatant following solids settling could not be removed from the centrifuge cone without disturbing the settled solids, the mass of the settled solids (M_{ss}) could not be measured directly. Therefore, the mass of the settled solids was determined by first calculating the mass of the supernatant following solids settling in the centrifuge cone using the measured supernatant density and volume ($M_{sl} = D_{sl}$ x V_{sl}), then subtracting this mass for the mass of the bulk slurry to get the mass of the settled solids ($M_{ss}=M_B-M_{sl}$). The density of the settled solids was then calculated ($D_{ss}=M_{ss}/V_{ss}$), as well as the wt% settled solids ($Wt\%_{ss}=M_{ss}/M_B$ x 100%).

Table 3.12. Density Measurements for Samples of AN-107 Diluted Feed (AN-107 PT), Sr/TRU Precipitated Slurry (DF-05 and DF-06), and Diluted Slurry (DF-13)

	Density, g/ml				
	Slurry	**Settled Solids**	**Supernatant Following Solids Settling**	**Centrifuged Solids**	**Centrifuged Supernatant**
AN-107 PT	1.32	1.47	1.38	1.88	1.311
AN-107 PT Duplicate	1.32	1.30	1.26	1.94	1.317
AN-107 PT Average	1.32	1.38	1.32	1.91	1.314
Relative % Difference	0%	12%	9%	3%	0.4%
DF-5	1.25	1.23	1.28	1.36	1.290
DF-5 Duplicate	1.26	1.31	1.26	1.34	1.298
DF-5 Average	1.26	1.27	1.27	1.35	1.294
Relative % Difference	1%	6%	2%	1%	0.6%
DF-6	1.26	1.37	1.29	1.42	1.291
DF-6 Duplicate	1.26	1.33	1.25	1.31	1.275
7-DF-6 Average	1.26	1.35	1.27	1.37	1.283
Relative % Difference	0%	3%	3%	8%	1%
DF-13	1.20	1.23	1.22	1.26	1.242
DF-13 Duplicate	1.20	1.21	1.23	1.32	1.250
DF-13 Average	1.20	1.22	1.22	1.29	1.246
Relative % Difference	0%	2%	1%	5%	0.6%

Table 3.13. Weight Percent and Volume Percent Solids measurements for samples of AN-107 Diluted Feed (AN-107 PT), Sr/TRU Precipitated Slurry (DF-05 and DF-06), and Diluted Slurry (DF-13)

	Wt% Settled[(1)]	**Wt% Centrifuged[(1)]**	**Vol% Settled[(1)]**	**Vol% Centrifuged[(1)]**	**Wt% Dissolved[(1)]**
AN-107 PT	7.0	4.5	6.3	3.2	41
AN-107 PT Duplicate	6.6	4.6	6.7	3.1	42
AN-107 PT Average	6.8	4.6	6.5	3.2	42.0
Relative % Difference	6%	2%	6%	3%	2%
DF-5	39	13	39	12	35
DF-5 Duplicate	40	13	39	12	36
DF-5 Average	40	13	39	12	36
Relative % Difference	2%	0%	0%	0%	3%
DF-6	43	15	40	13	43
DF-6 Duplicate	40	13	38	12	39
DF-6 Average	42	14	39	12	41
Relative % Difference	7%	14%	5%	8%	10%
DF-13	42	9.7	41	9.3	30
DF-13 Duplicate	42	9.6	41	8.8	30
DF-13 Average	42	9.6	41	9.0	30
Relative % Difference	0%	1%	0%	6%	0%

(1) Values include interstitial liquid.

The supernatant following solids settling was then added back to the centrifuge cones and centrifuged at approximately 1000 times the force of gravity for 1 hour. All of the centrifuged supernatant was then transferred to a graduated cylinder and its mass (M_{cl}) and volume (V_{cl}) recorded, and the density was calculated ($D_{cl}=M_{cl}/V_{cl}$). The mass (M_{cs}) and volume (V_{cs}) of the centrifuged solids were then recorded, and the density was calculated ($D_{cs}=M_{cs}/V_{cs}$). In addition, the wt% centrifuged solids ($Wt\%_{cs}=M_{cs}/M_B$ x 100%), and vol% centrifuged solids ($Vol\%_{cl}=V_{cl}/V_B$ x 100%) were also calculated.

The centrifuged solids and supernatants were then each dried at 105°C for 24 hours. The mass of the dried centrifuged supernatant (M_{dcl}) and dried centrifuged solids (M_{dcs}) were then measured. Assuming all mass lost during the drying process is water and not another volatile component, the weight percent total solids in the bulk slurry was calculated ($Wt\%_{ts} = (M_{dcs}+M_{dcl})/(M_{cs}+M_{cl})$ x 100%).

The results in Table 3.12 show the initial density of the diluted feed was 1.32 g/mL. Following the Sr/TRU removal treatment, the density decreased to 1.26 g/mL. The final diluted slurry, after the additional 19.24 wt% dilution, had a density of 1.20 g/mL. This value is very close to that calculated for this amount of dilution, 1.22 g/mL ([1.26+0.19]/[1.00+0.19] =1.218).

The density of the centrifuged solids in the initial diluted feed was 1.91 g/mL (entrained solids). The density of the Sr/TRU precipitate and entrained solids decreased to an average of 1.36 g/mL for samples DF-05 and DF-06. It then decreased again to 1.29 g/mL following the final dilution. The relative percent differences between duplicates were low, with the exception of 8% for sample DF-06. This drop in density with the final dilution (between DF-05/DF-06 and DF-13) indicates that dilution was inducing a change in the solid species, either decreasing the packing efficiency, dissolving the denser species (entrained solids), or both.

From the data in Table 3.13, there were 4.6 wt% centrifuged solids (including interstitial liquid) in the initial diluted feed. Following the Sr/TRU precipitation, the solids content increased to an average of 14 wt% for samples DF-05 and DF-06. Following the final dilution, the centrifuged solids content dropped to 9.6 wt%. This decrease to 9.6 wt% would be anticipated for a 19.24% dilution (14g/(126g+19.24g)*100%= 9.6%). The wt% total solids (including dissolved solids) was 42 wt% in the initial diluted feed. Following the Sr/TRU precipitation and final dilution this value was 30 wt%. Based on this 30 wt% value, a value of 44 wt% would be anticipated for samples DF-05 and DF-06. However, the highest measured value was 43% with an average of only 39 wt%. It is unclear why the wt% total solids for samples DF-05 and DF-06 were low, but the relative percent difference between duplicates was high as well, 3% and 10%, respectively. It is also possible that the value of 30 wt% for sample DF-13 was high, but both duplicates yielded the same result.

An additional calculation was performed to determine the wt% solids in the samples excluding all interstitial liquid (wt% undissolved solids). The wt% undissolved solids can be thought of as the solids left if all the supernatant could be removed from the bulk slurry. The following equation was used:

$$Wt\%\ undissolved\ solids = \left(1 - \frac{1 - \dfrac{M_{dsc}}{M_{cs}}}{1 - \dfrac{M_{dcl}}{M_{cl}}}\right) \times \frac{M_{cs}}{M_B} \times 100\%$$

This calculation assumes 1) that the supernatant and the interstitial liquid have the same composition, and 2) that all mass loss during the drying of the centrifuged solids is water loss from interstitial liquid. The results of this calculation are listed in Table 3.14 along with the wt% dried residue from the centrifuged solids (Solids Residue=M_{cs}/M_{dcs} x 100%), and dried centrifuged supernatant (Supernatant Residue= M_{dsl}/M_{sl} x 100%).

Table 3.14 shows the wt% undissolved solids for the duplicate initial diluted feed samples were 0.56 wt% and 0.67 wt%. This yields an average value of 0.62 wt% entrained solids in the diluted feed with a relative percent difference of 18%. This relative percent difference of 18% is not unreasonably high given the low solids content. The duplicates for the final diluted slurry (DF-13) also showed good reproducibility, 1.67 wt% and 1.58 wt% with an average of 1.62 wt% and a relative percent difference of 6%. However, the Sr/TRU precipitated samples, DF-05 and DF-06, showed a great deal of scatter with relative percent differences of 114% and 180%, respectively. It is possible that the low relative percent difference for the initial and final samples was fortuitous. However, it is more likely that solids inhomogeneity or a subsampling error was the cause of the variability in this analysis. While these are the most likely explanations, the issue is still not clear, since the remaining solids results in Table 3.12 and 3.13 showed reproducibility between the duplicate subsamples. Given a value of 1.62 wt% for the final diluted slurry, a value for the initial precipitated slurry of 1.93 wt% (1.62*1.19=1.93) would be anticipated. If we use the initial entrained solids value of 0.62 wt% and add the expected mass for Sr/TRU precipitate, we estimate 1.94 wt% solids. This is very close to the wt% solids predicted earlier based on ICP chemical analysis of the washed solids, 1.85 wt%.

Table 3.14. Results of Wt% Residual Solids and Undissolved Solids Calculation Following Drying at 105°C for 24 Hours for Samples of AN-107 Diluted Feed (AN-107 PT), Sr/TRU Precipitated Slurry (DF-05 and DF-06), and Diluted Slurry (DF-13)

Sample	Wt% Residual Centrifuged Solids	Wt% Residual Centrifuged Supernatant	Wt% Undissolved Solids
AN-107 PT	50.5	43.5	0.56
AN-107 PT Duplicate	51.7	43.4	0.67
AN-107 PT Average	-	-	0.62
Relative % Difference	-	-	18%
DF-5	67.1	33.3	6.6
DF-5 Duplicate	44.9	36.0	1.8
DF-5 Average	-	-	4.2
Relative % Difference	-	-	114%
DF-6	43.8	43.6	0.06
DF-6 Duplicate	44.6	38.9	1.21
DF-6 Average	-	-	0.64
Relative % Difference	-	-	180%
DF-13	41.6	29.6	1.67
DF-13 Duplicate	41.4	29.9	1.58
DF-13 Average	-	-	1.62
Relative % Difference	-	-	6%

3.6 Rheological and Flow Properties

Examples of typical rheograms for the initial AN-107 diluted feed and Sr/TRU precipitated waste are presented in Figures 3.7 and 3.8. All of the rheograms for the standards, samples, and duplicates are included in Appendix C. As seen in these examples, the slurry exhibits Newtonian behavior, as there is a nearly linear relationship between shear stress and shear rate over the shear rate range examined and no detectable yield stress. Since the viscosity is the ratio of the shear stress to the shear rate, the viscosity was nearly constant over the shear rate range examined for all samples. The viscosity of the initial diluted feed was between 10-12 cP, and the viscosity of the Sr/TRU precipitated slurry was between 7-18 cP.

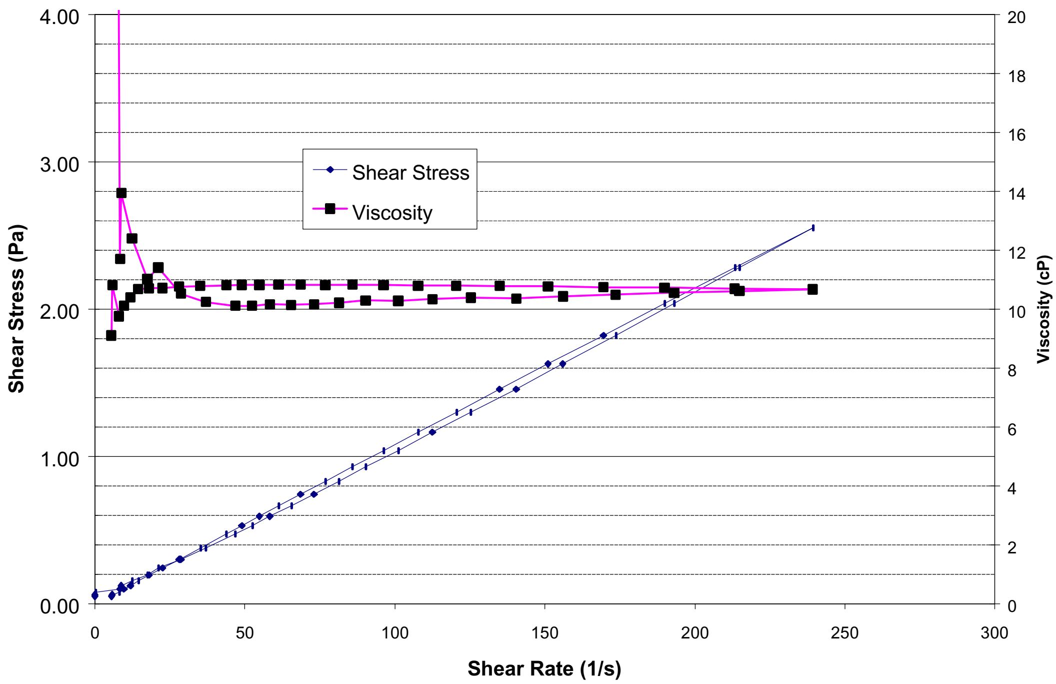


Figure 3.7. Initial AN-107 Diluted Feed: Sample 1

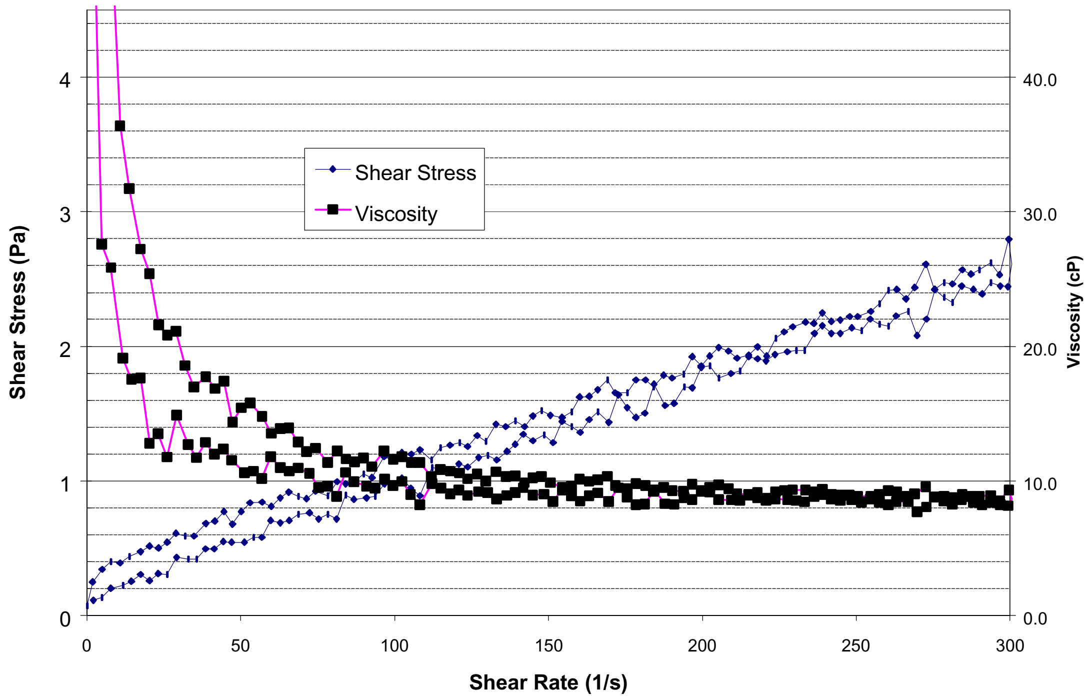


Figure 3.8. AN-107 Sr/TRU Precipitated Slurry: Sample 1 Analysis 1

4.0 CONCLUSIONS AND RECOMMENDATIONS

The new processing scheme for Sr/TRU removal involving precipitation by added strontium and permanganate worked well. Very high decontamination of Sr and TRU in AN-107 waste was accomplished with added concentrations of 0.075M Sr and 0.05M permanganate. These results support the use of lower concentration of reagent additions than used in this study. Optimization studies should be conducted to examine the reduction in added hydroxide from 1M to 0.5 M, reduction of Sr from 0.075M to 0.05M, and reduction in permanganate from 0.05M to 0.03M. The 4-hour digestion at 50°C appears unnecessary to obtain high decontamination of Sr and TRU using this treatment scheme. Adequate DFs were obtained after addition and thorough mixing of the reagents before the solids were digested. Mixing the precipitate for 1-hour at ambient temperature should be examined in future studies. However, these data indicate that thermodynamic equilibrium with the Sr/TRU precipitated solids was not reached during the processing time involved. Longer contact times may be required to allow equilibrium to be reached and reduce the possibility of the formation of solids after the filtration process.

The combined entrained solids and Sr/TRU precipitate was successfully filtered in the single element, crossflow filtration unit. At the preferred operating conditions, transmembrane pressure (TMP) of 50 psid and crossflow velocity of 12.2ft/sec, the average filtrate flux was 0.03 gpm/ft^2 or 1.75 m^3/m^2/day and the permeability was 0.53 m/day/bar. The filtrate flux was not as high as earlier tests with an archived AN-107 sample (0.11 gpm/ft^2), but this result was expected because of the higher concentration and presence of settled solids in the AN-107 diluted feed. The combined entrained and Sr/TRU removal solids content of the waste slurry was quite low, 1.9 wt%. The waste could only be dewatered approximately 50% to a solids content of approximately 4 wt% because of the large minimum operating volume of the CUF (~700-mL) and the limited amount of solids. The vacuum, air-dried solids were 36.6 wt% solids (63.4 wt% moisture). It appears that it will be difficult to dewater the combined entrained solids and Sr/TRU precipitate to the target value of >20 wt% solids by crossflow filtration. Solids washing in the CUF may allow higher solids levels but would require larger tank samples be treated for the CUF experiment, approximately 7-L samples of waste. Future tests should be conducted with larger tank waste samples to allow the determination of performance of the filter to higher solids content and evaluate the impact of washing in the CUF system.

The viscosity of the Sr/TRU precipitated slurry (1.9 wt% solids) was between 7-18 cP. The slurry showed a nearly linear relationship between shear stress and shear rate over the shear rate range examined and no detectable yield stress, i.e., Newtonian behavior.

The performance of the CUF system degraded with processing time. Frequent backpulsing could help keep average filtrate flux rates higher but could not recover or prevent degradation of filter performance with time. Rinsing and backpulsing the system with clean water was not adequate to restore filter flux. After some period of time, the unit/filter will require chemical cleaning such as the 1M nitric and 0.2M citric acid used for these studies. An external filter was also used to reduce the time and volume of water required for the final cleanup up the system. Additional studies need to be conducted to evaluate options for filter element cleaning, and impact to design if nitric/citric acid is required for filter cleaning.

The chemical decontamination of AN-107 waste with added Sr and permanganate is very rapid and occurs at room temperature in a matter of minutes. The filterability of the waste slurry and potential for post filtration precipitation will likely be the determining factor for the Sr/TRU removal process conditions. The waste as received from the tank is not filterable since the entrained solids rapidly foul the filter element. The Sr/TRU removal treatment results in a waste that can be filtered, the permanganate treatment greatly increasing the filterability by crossflow filtration. The filtration is still difficult with lower than expected flux rates, the filter element performance reduces with time, and the level of solids concentration impact of solids washing is unknown. Additional filtration studies need to be conducted and processing schemes involving solids settling/decant, precipitate recycle, and/or alternate dewatering should be considered.

5.0 REFERENCES

Brooks, K. P., P. R. Bredt, G. R. Golcar, S. A. Hartley, M. W. Urie, J. M. Tingey, K. G. Rappe, L.K Jagoda. 1999. *Ultrafiltration and Characterization of AW-101 Supernatant and Entrained Solids*, PNWD-3000, Battelle, Richland, Washington.

Brooks, K. P., P. R. Bredt, G. R. Golcar, S. A. Hartley, L.K. Jagoda, K. G. Rappe, M. W. Urie. 2000. *Characterization, Washing, Leaching, and Filtration of C-104 Sludge*, PNWD-3024, Battelle, Richland, Washington.

Hallen, R. T., S. A. Bryan, and F. V. Hoopes. 2000a. *Development of an Alternative Treatment Scheme for Sr/TRU Removal: Permanganate Treatment of AN-107 Waste*, PNWD-3047, Battelle, Richland, Washington.

Hallen, R. T., K. P. Brooks, and L. K. Jagoda. 2000b. *Demonstration of Entrained Solids and Sr/TRU Removal Processes with Archived AN-107 Waste*, PNWD-3033, Battelle, Richland, Washington.

Hendrickson, D. W. 1997. *Hanford Complexant Concentrate and Salt Cake Cesium Removal Using Superlig® 644.* SESC-EN-RPT-007 Rev. 0, SGN Eurisys Services Corporation, An SGN/Cogema, Inc. Company.

Krot, N. N., V. Shilov, A. Bressonov, N. Budantseva, I. Charushnikova, V. Perminov, and L. N. Astafurova. 1996. *Investigation on the Coprecipitation of Elements from Alkaline Solutions by the Method of Appearing Reagents*, WHC-EP-0898, Westinghouse Hanford Company, Richland, Washington.

G.J. Lumetta and F.V. Hoopes. 1999. *Washing of the AN-107 Entrained Solids*, PNWD-2469, Battelle, Richland, Washington.

Orth, R. J., A. H. Zacher, A. J. Schmidt, M. R. Elmore, K. R. Elliott, G. G. Neuenschwander, and S. R. Gano. 1995. *Removal of Strontium and Transuranics from Hanford Tank Waste via Addition of Metal Cations and Chemical Oxidant - FY 1995 Test Results*, PNL-10766, Pacific Northwest Laboratory, Richland Washington.

Rapko, B.M., G.J. Lumetta, and M.J. Wagner. 1996. *Oxidative Dissolution of Chromium from Hanford Tank Sludges Under Alkaline Conditions*, PNNL-11233, Pacific Northwest National Laboratory, Richland, Washington.

Rapko, B.M. 1998. *Oxidative Alkaline Dissolution of Chromium from Hanford Tank Sludges Results of FY 98 Studies*, PNNL-11908, Pacific Northwest National Laboratory, Richland, Washington.

River Protection Project Waste Treatment Plant (RPP-WTP) (formerly TWRS) Privatization Contract. 1996. DE-RP06-96RL13308. U.S. Department of Energy, Richland, Washington.

Schroeder, Norman C., *Oxidation, Characterization, and Separation of Non-Pertechnetate Species in Hanford Wastes,* TTP Number AL17C321, Los Alamos National Laboratory, Los Alamos, New Mexico.

SRTC 1997a, *Hanford Envelope Archive C Tank Waste Precipitation Study*, March 20, 1997, SRTC-BNFL-004, revision 0, Savannah River Technology Center, Aiken, South Carolina.

SRTC 1997b, *Hanford Simulant Tank Waste Precipitation Study*, July 8, 1997, SRTC-BNFL-006, revision 1, Savannah River Technology Center, Aiken, South Carolina.

SRTC 1997c, *Hanford Envelope C Tank Waste Precipitation Study*, August 13, 1997, SRTC-BNFL-005, revision 2, Savannah River Technology Center, Aiken, South Carolina.

SRTC 1997d, *Hanford Envelope C Tank Waste Sr/TRU Precipitation Demonstration*, November 18, 1997, SRTC-BNFL-024, revision 0, Savannah River Technology Center, Aiken, South Carolina.

Townson, Paul S., 1999, *Sr/TRU Precipitation and Ultrafiltration Test Specification, TSP-W375-99-00004 Rev. 1,* August 17, 1999, BNFL Inc., Richland, Washington.

Urie, MW, J. J. Wagner, L. R. Greenwood, O. T. Farmer, S. K. Fiskum, R. T. Ratner, and C. Z. Soderquist. 1999a. *Inorganic and Radiochemical Analysis of AW-101 and AN-107 Tank Waste,* PNWD-2462, Battelle, Richland, Washington.

Urie, M. W., J. J. Wagner, L. R. Greenwood, O. T. Farmer, S. K. Fiskum, R. T. Ratner, and C. Z. Soderquist. 1999b. *Inorganic and Radiochemical Analysis of AW-101 and AN-107 'Diluted Feed' Materials,* PNWD-2463, Battelle, Richland, Washington.

APPENDIX A: TEST INSTRUCTION AND DATA SHEETS

Sr/TRU Precipitation of AN-107 Diluted Feed and Combined Removal of Entrained Solids and Sr/TRU Precipitate by Crossflow Filtration

Data Sheets

Log Book Entries

APPENDIX B: ANALYTICAL DATA

APPENDIX C: RHEOGRAMS

APPENDIX D: CALCULATIONS

APPENDIX E: STAFF AND ROLE/RESPONSIBILITY

Staff Member	Role/Responsibility
Richard Hallen	Scientist/Technical Leader - Sr/TRU Precipitation
Paul Bredt	Scientist/Physical and Rheological Properties
Kriston Brooks	Engineer/CUF System and Solids Removal
Lynette Jagoda	Engineer/CUF System Cleaning
Don Rinehart	Technician/Hot Cell Tests-Sr/TRU PPT/CUF Operation
Ralph Lettau	Technician/Hot Cell Tests-Sr/TRU PPT/CUF Operation
Dave Ortiz	Technician/Hot Cell CUF Operation and Cleaning
Vaughn Hoopes	Technician/Hot Cell CUF cleaning and sample prep.
Mike Mann	Technician/Hot Cell Filtrate Composite
Mac Zumhoff	Technician/Hot Cell Operations

Distribution

No. of Copies

OFFSITE

2 DOE/Office of Scientific and Technical Information

No. of Copies

ONSITE

5 British Nuclear Fuels, Limited

M. E. Johnson (4)	BN-FL
A. Thompson	BN-FL

11 Pacific Northwest National Laboratory

P. R. Bredt	P7-25
K. P. Brooks	K6-24
R. T. Hallen	K2-12
L. K. Jagoda	K6-24
D. E. Kurath	P7-28
E. V. Morrey	P7-28
Technical Report Files (5)	K1-06

APPENDIX A: TEST INSTRUCTION AND DATA SHEETS

Sr/TRU Precipitation of AN-107 Diluted Feed and Combined Removal of Entrained Solids and Sr/TRU Precipitate by Crossflow Filtration

Data Sheets

Log Book Entries

PNNL Test Instruction

Document No.: BNFL-TI-29953-052
Rev. No. 0

Title: Sr/TRU Precipitation of AN-107 Diluted Feed and Combined Removal of Entrained Solids and Sr/TRU Precipitate by Crossflow Filtration

Work Location: RPL SFO HLRF	**Page** 1 of 15
Author: Richard T. Hallen	**Effective Date:** Upon Final Approval **Supercedes Date: New**
Use Category Identification: Information	
Identified Hazards: __ Radiological __ Hazardous Materials __ Physical Hazards __ Hazardous Environment __ Other:	**Required Reviewers:** X Technical Reviewer __ SFO Manager

Are One-Time Modifications Allowed to this Test Instruction? X Yes __ No

NOTE: If Yes, then modifications are not anticipated to impact safety. For documentation requirements of a modification see SBMS or the controlling Project QA Plan as appropriate.

On-The Job Training Required? ____ Yes or X No

FOR REVISIONS:
Is retraining to this procedure required? ____ Yes X No

Does the OJT package associated with this procedure require revision to reflect procedure changes?
____ Yes ____ No X N/A

Approval	Signature	Date
Author	Richard T. Hallen	9/16/99
Technical Reviewer	RJ Brooks	9/16/99

UnControlled Document

1.0 Applicability

This test instruction is to be used to perform studies on permanganate treatment for Sr/TRU removal and precipitate removal from AN-107 diluted feed. The entrained solids will not be removed prior to Sr/TRU precipitation. The combined entrained solids and Sr/TRU precipitate will be removed using the Cell Unit Filter (CUF) in the HLRF A-cell from approximately 1.4-L of AN-107 diluted feed.

2.0 Supporting Documents

This test instruction is not a stand-alone document. It will be used in conjunction with PNNL Operating Procedure BNFL-TP-29953-020 which contains the necessary procedural information for the safe operation of the CUF. It is also linked to PNNL Test Plan BNFL-TP-29953-013 Rev. 1 which contains an overall description of the goal of this effort, ES&H compliance, emergency response, and the hazards assessment and mitigation.

3.0 Responsible Staff

The staff responsible for executing this test plan are as follows.

- Task Managers – Rich Hallen
- SFO Manager – Randy Thornhill
- Test Scientists/Engineers – Kriston Brooks, Lynette Jagoda, and Rich Hallen
- Hot Cell Technician –Don Rinehart, and Ralph Lettau
- Radiological Control Technician

4.0 Materials, Equipment, Supplies and Reagents Needed

4.1 Materials Required

1. Twenty 20 mL glass scintillation vials for filtrate and slurry samples, pre-labeled on top and side as follows: DF-01 through DF-20.
2. Four 1 liter polyethylene bottles. They should be labeled as follows: "AN-107 DF CUF Filtrate," "AN-107 DF Filtrate (filter unit receiver)," "CUF Dewatered Slurry," and "CUF AN-107 DF First Rinse."
3. Four 100 mL bottles for each wash solution labeled wash 1 through 4. These can be filter unit bottles if small ones are available.
4. Two 10 liter containers, one labeled for the alkaline rinses and the other labeled for the acidic rinses.
5. Containers for draining from the bottom of the pump and from the sample valve.
6. 12 liters of 0.2 micron filtered DI water for determining clean water flux and for rinsing the CUF

4.2 Equipment

1. 4000 gram balance
2. pH paper
3. Hand held camera. To be used to read filtrate flowmeter.

4. Stopwatch
5. Calculator
6. CUF Ultrafiltration system with 100 mL plug in place using 0.1 micron Mott-L filter and new pump rotor
7. 1000 W Chiller
8. 4-L flask (large funnel for waste addition)
9. hot-stir plate
10. big stir bar
11. syringe/tube for sampling 4-L flask
12. 1-L deadend filtration unit and four 150 mL units
13. 5-mL pipette (4 needed)
14. 10 mL volumetric flask for density determination (ball flask)

4.3 Reagents Needed

1. 1 liters of 1M HNO_3 + ~0.1M Citric Acid
2. 1 liter of 5 ppm hypochlorite solution (pH > 7)
3. 264.6 mL of 3.5185M NaOH
4. 96.6 mL of 1.0M NaMnO4
5. 142.8 mL of 1.0M Sr(NO3)2
6. 250 mL of 0.01M NaOH (wash solution)

4.4 Other Supplies

1. Workplace Copy of Operating Procedure BNFL-TP-29953-020
2. Extra Copies of Data Sheets 1, 2, and 3
3. Laboratory Record Book
4. DAS disk for recording data

5.0 Test Instructions for CUF Operation and Permanganate Treatment

The laboratory record book (LRB) shall be used to record other testing information as required by this procedure and all test conditions not stated by this procedure.

Cross-contamination between samples and contamination of samples from outside sources must be minimized at each step. Use new tools and bottles for each sample as much as possible. Those tools that are reused should be washed and rinsed prior to reuse.

Keep all test materials in sealed containers as much as possible to prevent them from drying.

5.1 Pre-start for Sr/TRU Precipitation and CUF Operation

5.1.1 Inventory materials, equipment, supplies, and reagents to ensure all required items are available. Assure that all materials have been modified for remote handling.

5.1.2 Do the following and initial and date when each item is completed.

_________ Review PNNL Operating Procedure BNFL-TP-29953-020.

BDJ Review the work instructions in BNFL-TI-29953-052.

5.1.3 Conduct the "0.0 Pre-Start" operations in BNFL-TP-29953-020. Drain the system overflow container. 6:00 AM Valve 6 check - operate easily

?pt run first 5.2

5.1.4 Perform "1.0 Start-Up" operations in BNFL-TP-29953-020 with 1 liter of filtered DI water with one variation: V4 the filtrate control valve should be closed. Run CUF for 5 minutes at between 4-6 gpm. In-line pressure should be varied from using V1 from 10 to 70 psig. Ensure that there are no leaks in the system. If leaks are detected, shut down system immediately.

5.1.5 Conduct the "10.0 Draining the system" operation in BNFL-TP-29953-020.

5.1.6 Perform "1.0 Start-Up" operations in BNFL-TP-29953-020 with 1.0 liter of filtered, distilled water with one variation: V4 the filtrate control valve should be closed. Run CUF for 5 minutes at between 4-6 gpm. In-line pressure should be varied from using V1 from 10 to 70 psig.

5.1.7 Conduct the "10.0 Draining the system" operation in BNFL-TP-29953-020.

5.1.8 Perform "1.0 Start-Up" operations in BNFL-TP-29953-020 with 1.0 liter of filtered, distilled water

5.1.9 Perform "6.0 Back pulsing" operations in BNFL-TP-29953-020.

5.1.10 Shut off the system and conduct the "10.0 Draining the system" operation in BNFL-TP-29953-020.

5.1.11 Perform "1.0 Start-Up" operations in BNFL-TP-29953-020 with 1.0 liter of filtered, distilled water.

5.1.12 **Determine clean water flux.** Conduct the "3.0 Operation during Ultrafilter Recycle Mode" operations in BNFL-TP-29953-020 using the conditions below. Filtrate flow rate should be monitored and data collected in the operating procedure. Each test should be performed for only 20 minutes and the system should be back pulsed. After each condition, the test engineer should initial and date the table below.

Condition	Flowrate (gpm)	Transmembrane Pressure (psig)	Initial and date when complete
1	4.20	10	
2	4.20	20	
3	4.20	30	

5.1.13 Shut off the system and conduct the "10.0 Draining the system" operation in BNFL-TP-29953-020. Drain as much as possible water out of the system. Be sure to drain the pump also.

5.2 Waste for Sr/TRU Removal Treatment

NOTE: The CUF clean water flux can be completed after Step 5.2 or while the waste is digesting (4 hours of heating)

5.2.1 Check balance calibration and record information below.

M&TE List:

RTK Balance 1: Mettler PM 400
Calib ID 362-06-01-061 Calib Exp Date 8/2000
Location A cell South

RTK Balance 2: Mettler PM 4000
Calib ID 362-06-01-054 Calib Exp Date 8/2000
Location A cell North

Thermocouple:
Calib ID TC325-427 Calib Exp Date 9-2001
Location ______ Thermocouple type K 0-100°C

Digital Thermometer: Fluke
Calib ID S/N 2275 Calib Exp Date 2/2000
Location A cell south

5.2.2 Setup the ppt reaction equipment as specified. Record the tare weight of the reaction flask and the stir bar. This may be done outside the hot cell. The precipitation is to be conducted in a 4-L flask. Secondary containment should be used to allow recovery from a possible breach of the 4-L flask. Place stir bar in flask.

Hot cell temp 29.6

Tare weight of reaction flask ______
Tare weight of stir bar 38.4144

Not all of the AN-107 Diluted Feed is to be used for this test. Approximately 400 mL should be saved for future Sr/TRU optimization studies. Record the weights of waste/jar(s) below. Swirl/mix the jar(s) of waste thoroughly. Transfer 1.4-L or 1848 grams of waste to the reaction flask. (The supernate density is reported as 1.32 g/mL.) There should be some solids that may have settled. Jar UFE is reported to have some ¼ inch chucks of solids. If solids are observed, record observation (record observations in the LRB).

Jar	Tare, g	Total, g	Sludge, g	volume, mL	Identify Jars Used
AN-107 UFA	356.65	738.27	381.62	289.10	use
AN-107 UFB	351.27	915.85	564.58	427.71	use
AN-107 UFC	357.33	919.98	562.65	426.25	Save
AN-107 UFD	356.68	922.60	565.92	428.73	use
AN-107 UFE	358.14	706.10	347.96	263.61	use
		Sum Total	2423 g	1835 mL	

5.2.3 Record the weights of the jars with lid in the spaces provided below. Pour waste into reaction flask. I large funnel may be necessary to prevent spill any waste since the jars are near full. Also, if large chucks of solids are present, use a piece of plastic screen in the funned to remove the solids. Calculate the amount of material transferred to the reaction flask.

Jar	Jar + Waste	Empty Jar	Waste, g	volume, mL
AN-107 UFA	737.85	360.053	377.797	
AN-107 UFB	915.36	354.584	560.776	
AN-107 UFC				
AN-107 UFD	922.26	359.417	562.843	
AN-107 UFE	705.29	361.268	344.022	
		Sum Total	1845.438	

If large quantities of solid remain, the 3.5M NaOH solution may be used to rinse out jars and rinse down funne RTM

Reported Density = 1.32 g/mL   Total volume added 1398.06 mL

5.3 Permanganate Treatment of AN-107 Diluted Feed

5.3.1 Turn on stirrer. Turn on the temperature recording devise. Record temperatures of cell and waste. Flask TC reads 31.9 before waste added

5.3.2 While continuously stirring the waste sample, personnel are to slowly (over a 2 minute period) add 264.6 mL of 3.5185M NaOH. This is the entire content of the bottle labeled 3.5M NaOH.

Tare bottle of 3.5M NaOH 648.31   see prep sheet   264.16 mL
Tare Empty bottle 351.762 g
Weight Added 296.548 g

d =

5.3.3 While continuously stirring the waste sample, personnel are to slowly (over a 10 minute period) add 142.8 mL of 1.0 M $Sr(NO_3)_2$. This is the entire content of the bottle labeled 1M Sr.

Tare bottle of 1M Sr 397.19   see prep sheet
Tare Empty bottle 231.823 g
Weight Added 165.367 g   142.74 mL

5.3.4 While stirring, slowly (over a 10 minute period) add 96.6 mL of 1 M $NaMnO_4$. This is the entire content of the bottle labeled 1M NaMnO4

96.46 mL added

Tare bottle of 1M NaMnO4 334.88 See prep sheet
Tare Empty bottle 229.322 g
Weight Added 105.558 g

5.3.5 Allow the waste to thoroughly mix after addition of all of the reagents, i.e. stir for 30 minutes. After 30 minutes collect 40 mL of slurry in a bottle. (Put this aside and turn heater on.) then while heating, use a 10-mL volumetric flask (ball flask) to determine the slurry density for duplicate samples. Pour contents back into the bottle. Filter two, 20 mL samples in vials DF-01 and DF-02 for chemical analyses. Record the weight and sample number in Data Sheet 3.

DF-01 - filtered
DF-02 - NOT Filtered

Tare flask 9.177 g
Flask plus 10 mL of slurry 22.147 g
Weight of 10 mL slurry 12.970 g
density of slurry 1.297 g/mL

Tare flask 9.372
Flask plus 10 mL of slurry 22.354 g
Weight of 10 mL slurry 12.982 g
density of slurry 1.298 g/mL

5.3.6 Heat the waste mixture at 50 ± 5°C with stirring for 4 hours. Thermocouple reads 47.5°C at 50°C RJW

5.3.6 Turn off the stirrer and allow the waste to cool to 25 ± 5°C. If possible, use a video recorder to document the settling behavior of the waste mixture. Record the volume of settled solids if possible. ____________ mL

5.3.7 Collect and filter two 20 mL samples for transfer to SAL for chemical analyses in vials DF-03 and DF-04. Record the weight and sample number in Data Sheet 3. These samples need to be filtered with 0.45 um syringe filter, and density of the filtrate determined. Then submitted for chemical analyses.

Samples could not be easily filter in Hot cell

Tare flask ____________
Flask plus 10 mL of filtrate ____________
Weight of 10 mL filtrate ____________
density of filtrate ____________ g/mL

Tare flask ____________
Flask plus 10 mL of filtrate ____________
Weight of 10 mL filtrate ____________
density of filtrate ____________ g/mL

5.4 Completion of Permanganate Treatment/Precipitation and Startup of CUF Testing

(The CUF testing is to occur as soon after precipitation as possible. If for some reason, a delay is required contact the task manager for further instruction, work 375-6919 or home 943-5846.)

5.4.1 Verify that the M&TE List has been completed (and are working).

- Check valve 6 on CUF to make sure it is operational before adding slurry to CUF reservoir RJA

5.4.2 Conduct the "1.0 Start-Up" operations in BNFL-TP-29953-020 using permanganate treated AN-107. Remove the stir bar. Record the weight of the reaction vessel and ppt. Swirl/mix the waste slurry thoroughly before transferring to the CUF slurry reservior. There will be some solids left in the flask but try to minimize these by swirling the flask during the transfer of the final small volumes of wastes. If excessive solids remain, consult with the cognizant scientist/engineer or task leader on recovering these solids. A small amount of supernate could be pipetted from the slurry reservoir to wash these solids out of the flask. Record the method of recovery on this test instruction or in the LRB.

5.4.3 Record the weights of waste and reaction flask. - * Rich Hallen already Pulled 2 samples before this wt.

Vessel and ppted AN-107 3609.34 g
Empty vessel 1320.32 g

Weight transferred to CUF 2289.02 g

5.4.4 Record the level in the slurry reservoir sight glass.

Height Not taken inches

5.5 CUF Operation: Sr/TRU Precipitate Removal Test with AN-107 Diluted Feed

* This needs to be completed after 8:00 AM so Paul C run rheology RJA

5.5.1 Obtain slurry samples following "7.0 Slurry Sampling" in BNFL-TP-29953-020. The samples should be taken before any filtration tests. The first two slurry samples should not be saved, but dumped back into the slurry reservoir. Allow the waste to recirculate in the CUF for 5 minutes with no filtration, i.e. the CUF pump in operation, and the throttle valve completely open (no pressure or filtrate flux). While the waste is recirculating, use the slurry sampling value to collect two 40-mL slurry samples and conduct rheological measurements. Transfer the waste back to the slurry reservoir. Collect two 20-mL slurry samples in vials DF-05 and DF-06. Use one for physical property measurements and store the other. Record the weight and sample number in ~~Data Sheet 3~~ Log Book ✓ RJA

use low flowrate 8-1-29 PM RJA

5.5.2 Conduct the "3.0 Operation during Ultrafilter Recycle Mode" operations in BNFL-TP-29953-020 using the conditions below. Filtrate flow rate should be monitored and data collected as specified in the operating procedure. After each condition, the test engineer should initial and date the table below. If no filtrate flow occurs or the filtrate flow is less than 10 mL/min, discontinue testing, back pulse, and move to the next condition.

- I suggest we collect a single filtrate sample during each condition RJA

NOTE: Test conditions below are suggested and conditions do not need to be run for the full 1 hour if filtrate flux drops to low values. Collect data at 10 minute intervals on data sheet.

F. trate only *

Condition	Flowrate (gpm)	Transmembrane Pressure (psig)	Initial and date when complete
1	(pull sample) 4.20	50	HB 9/20/99
2	4.20	30	HB 9/20/99
3	(pull sample) 4.20	70	HB 9/20/99
4	3.13	Optimum from 1-3	HB 9/20/99
5	(pull sample) 5.23	Optimum from 1-3	HB 9/21/99
6	pull sample 4.20	50	EJB 9/21/99
7	~~(pull sample)~~ Optimum from 1-6	Optimum from 1-6	skipped

* think plugg forming - pull another

5.5.3 Conduct dewatering in CUF. Follow procedure in BNFL-TP-29953-020. Conduct dewatering using condition 7. Collect the filtrate in the tared 1-L bottle labeled "AN-107 DF CUF Filtrate." Back pulse as necessary to keep filtrate flux to acceptable levels. The volume will only be reduced to ~800 mL in the CUF. Additional dewatering will be done using deadend filtration.

obtain density of CUF Filtrate using 10 mL volumetric flas

Tare weight of AN-107 DF CUF Filtrate Bottle 104.50
Bottle and filtrate 1297.76 Weight of CUF Filtrate 1193.26

5.5.4 Obtain two 20-mL filtrate samples following "8.0 Filtrate Sampling" in BNFL-TP-29953-020 and using the pre-labeled sample vials after approximately half of the dewatering in the CUF has been completed. Record the weight and sample number in Data Sheet 3. One will be used for chemical and radiochemical analyses and one stored.

5.5.5 Conduct the "11.0 Shutting down" operation in BNFL-TP-29953-020.

5.6 Draining the Dewatered Slurry from the CUF

5.6.1 Tare the 1 liter bottle labeled, "AN-107 DF CUF Dewatered Slurry."

Weight of bottle and lid 105.10 g

5.6.2 Conduct the "10.0 Draining the system" operation in BNFL-TP-29953-020. Collect slurry in 1 liter bottle. Make sure as much materials as possible has been collected. This material will be further dewatered using a deadend filter unit. Weigh bottle after all slurry has been removed from the CUF.

Weight of slurry, bottle and lid 987.94 g
Weight of material collected 882.84 g

Cell temp - 31 °C

NOTE: Proceed with rinsing the CUF and conduct 5.7 Dewatering using a Deadend Filtration Unit when time allows. This can start as soon as the initial rinse of the CUF is completed.

10 mL
tare flask 9.444 g
flask + filtrate 22.296 g
density 1.285

tare flask 9.449 g
flask + filtrate 23.308 g
density 1.286 g

first rinse completed
05:20 9/21/97

5.6.3 Conduct the "9.0 Rinsing the system" operation in BNFL-TP-29953-020. The first rinse should be done with 1 liter of distilled water. This liquid should be collected and saved in the container labeled "AN-107 DF CUF First Rinse." The second rinse should be done with ~~2~~ 1 liters of filtered, distilled water, and the final rinse with 1 liter filtered, distilled water. The second and third rinses should be collected separately from the first in the alkaline rinse storage container. 1 liter used

— Done —

NOTE: Once the CUF has been rinsed you may proceed to Section 5.7 and come back and complete the CUF cleanup later.

5.6.4 Perform "1.0 Start-Up" operations in BNFL-TP-29953-020 with 1.0 liter of filtered, distilled water.

5.6.5 Perform "6.0 Back pulsing" operations in BNFL-TP-29953-020.

5.6.6 Determine the clean water flux at 20 psid transmembrane pressure and flow rate of 4.2 GPM following "3.0 Operation during Ultrafilter Recycle Mode" operations in BNFL-TP-29953-020. Filtrate flow rate should be monitored and data collected in the operating procedure. Data should be collected for at least 20 minutes and the system should be back pulsed.

5.6.7 Shut off the system and conduct the "10.0 Draining the system" operation in BNFL-TP-29953-020. System did not clean up with water only

5.6.8 Conduct the acid wash of the CUF unit with 1M HNO3/0.1-0.2M Citric Acid as described in the CUF operating procedure if needed to regain clean water flux. The acidic solution should be allowed to sit in the CUF overnight. When drained, the acidic solution should be placed in a separate container.

5.6.9 The CUF should be drained according to "10.0 Draining the system" operation in BNFL-TP-29953-020 and rinsed at least 3 times with filtered, distilled water to bring the pH back up to neutral. The acidic solutions should be placed in a separate container from the alkaline ones.

5.6.10 Perform "1.0 Start-Up" operations in BNFL-TP-29953-020 with 1.0 liter of filtered, distilled water.

5.6.11 Conduct the "3.0 Operation during Ultrafilter Recycle Mode" operations in BNFL-TP-29953-020 using the conditions below. Filtrate flow rate should be monitored and data collected in the operating procedure. Each test should be performed for only 20 minutes and the system should be back pulsed. After each condition, the test engineer should initial and date the table below.

Condition	Flowrate (gpm)	Transmembrane Pressure (psig)	Initial and date when complete
1	4.20	10	
2	4.20	20	

3	4.20	30	

5.6.12 Shut off the system and conduct the "10.0 Draining the system" operation in BNFL-TP-29953-020.

5.7 Dewatering with the Deadend Filter Unit → filtrate flux low so recombine Filtrate and Slurry - dilute ~25% - rerun CUF matrix - see supplemental TI-052

5.7.1 Tare the 1-L receiver bottle/lid and filter assembly/lid of the filtration unit.

Tare of receiver bottle -band and lid 198.66 g
Tare of the filter assembly and lid 104.78 g

5.7.2 Assemble 1-L filtration unit and filter all of slurry in bottle labeled "CUF Dewatered Slurry." Solids should have settled on setting. Decant most of the supernate from the bottle and filter. The supernate should filter quite fast. When approximately half the material has been filtered, swirl the bottle to suspend the solids. Continue filtering the slurry. If excessive solids remain in the bottom of the bottle, small amounts of filtrate can be used to rinse the solids from the bottle. Record weight of empty bottle. Filter the solids until compacted on filter and no free liquid remains. Disassemble filter unit and weigh

Tare of receiver bottle/lid and filtrate ____________ Weight of filtrate ____________
Tare of the filter assembly/lid and wet solids ____________ Weight of wet solids ____________

5.7.3 The two 1-L bottles of filtrate, CUF filtrate and the deadend filtrate, should be transfer to SAL and combined to make a composite filtrate. Take duplicate samples of the filtrate composite. Record weight of each filtrate and vial used on Data Sheet 3.

5.8 Sr/TRU Solids Washing

Note: Additional filter units should be available because the filters may plug during washing.

per phone call with Mike Johnson 9/24/99 3:56PM RTA

5.8.1 The Sr/TRU solids are to be washed with four equal volumes of stabilized water (0.01M NaOH). Place the filter assembly containing wet solids on a tared receiver flask labeled wash 1. Estimate the volume of compacted solids on the filter. Use ~~3~~ 2 times the solids volume of wash solution ~~but not more then 60 mL.~~ Tare a 100 mL graduated cylinder. Add the volume of water. Record the weight. With the vacuum turned off, pour the wash water on to the wet solids, record weight of empty graduated cylinder. Allow the solids to rewet before turning the vacuum on. Then filter the solids until compacted on the filter and no free liquid remains. Disassemble and weigh receiver flask and filter assembly/solids.

Tare graduated cylinder ________ + ___mL of wash __________ weight of wash______
Tare of receiver bottle and lid ____________

So we will need more wash solution and a larger graduate measuring device, and larger wash solution bottles.

Tare of receiver bottle/lid and wash ____________ Weight of wash 1 ____________
Tare of the filter assembly/lid and washed wet solids ____________

5.8.3 Repeat 5.8.1 for second wash and record weights. (Wash 2)

Tare graduated cylinder _________ + ___mL of wash __________ weight of wash______
Tare of receiver bottle and lid ____________
Tare of receiver bottle/lid and wash ____________ Weight of wash 2 ____________
Tare of the filter assembly/lid and washed wet solids ____________

5.8.4 Repeat 5.8.1 for third wash and record weights. (Wash 3)

Tare graduated cylinder _________ + ___mL of wash __________ weight of wash______
Tare of receiver bottle and lid ____________
Tare of receiver bottle/lid and wash ____________ Weight of wash 3 ____________
Tare of the filter assembly/lid and washed wet solids ____________

5.8.5 Repeat 5.8.1 for fourth wash and record weights. (Wash 4)

Tare graduated cylinder _________ + ___mL of wash __________ weight of wash______
Tare of receiver bottle and lid ____________
Tare of receiver bottle/lid and wash ____________ Weight of wash 4 ____________
Tare of the filter assembly/lid and washed wet solids ____________

5.8.6 Using a pipette, transfer 5 mL from each of the 4 wash samples to separate vials (this volume does not have to be accurate) and transfer 5 mL of each wash solution into a single vial for a composite wash sample. Record the weight and sample number in Data Sheet 3 for each individual sample and for the composite sample. The individual wash samples will be analyzed by ICP only. The composite will be used for chemical and radiochemical analyses.

5.8.7 Tare a jar and transfer the wet solids out of the filter assembly. Record the weight of wet solids in the jar and the weight of the filter assembly after the solids have been removed. Dry the solids for at least 24 hours at 105 °C and record the weight of dry solids. Transfer 1 gram of solids to a 20 mL vial for chemical analyses. Record vial number and weight on Data Sheet 3.

Tare Jar ____________ Jar plus wet solids ____________ Weight of wet solids ____________
Jar and dry solids weight ____________ Amount of dry solids ____________

5.8.8 Retain all filtrate, wash solution, and solids for future testing.

5.10 Experimental Clean Up and Sample Disposition.

Clean up all of the equipment used.

Do not discard any samples without written instructions from the task manager. Duplicate samples were collected at all sampling points, but only primary samples submitted for analyses. The duplicates are to be retained until review of the analytical data is completed and notification is writing is received to dispose of these samples. The alkaline and acid CUF wash solutions should be disposed of properly.

6.0 Sample Analysis

The point of contact for the sample analysis of the filtrate, slurry, wash, and washed solids samples is Mike Urie and Rick Steele. Table 1 listed the analyses to be performed on the samples.

The first two slurry samples need to have slurry density determined. This can be during the tests or before analytical sample prep. Once the density is determined the samples should be filtered with a 0.45 micron disposable syringe filter as soon as possible (can be filtered in A-cell when taken if density determined). The digested ppt samples should also be filtered as soon as possible.

Only one sample of each wash and one wash composite will be collected and analyzed until the data can be reviewed. If any inconsistencies are seen, the original wash solutions will be resampled and analyzed again. The wash solutions will not be combined so they may be sampled individually or a composite sample remade.

Table 1. Samples Taken during Testing and Their Required Analyses

Process Step	Number of Samples	Sample Type	Step	Analysis	Analysis Description
Precipitated Feed (.45 micron filtrate)	2	Slurry Filtrate	After Precipitation (Before Digestion)	Physical Properties Chemical Analyses	slurry density Sr-90, ICP, AEA
Digested PPT (0.45um)	2 (store 1)	Filtrate	After Digestion	Physical Properties Chemical Analyses	filtrate density Sr-90, ICP, AEA
Recycled slurry	2 (40 mL) 2 (store 1)	Slurry Slurry	CUF sampled CUF sampled	Rheology Physical Properties	viscosity Table 4
Middle De-water Step	2 (store 1)	Filtrate	During Condition 2.8	Chemical Analyses	Table 2
Filtrate Composite	1	Filtrate	CUF + deadend	Chemical Analyses	Table 2
Four Washes	1 each	Filtrate	After Each Wash	Chemical Analyses	ICP Only (Na)
Wash Composite	1	Filtrate	After All Washes	Chemical Analyses	Table 2
Washed Solids	2	Dry Solids	Final Solids	Chemical Analyses	Table 2

Table 2. Analytical Requirements for Washed Solids, Filtrate, and Wash Solutions

Analyte	Washed Solids Minimum Reportable Quantity (MRQ) µCi/gm	Filtrate and Wash Solutions Minimum Reportable Quantity (MRQ) µCi/ml
Cesium-137	6.0E-02	9.0E+00
Strontium-90	7.01E+01	1.5E-01
Technetium-99	**6E+00 µgm/gm**	1.5E-03
Americium-241	1.2E-03	7.2E-04
Europium-154	6.0E-02	2.0E-03
Europium-155	6.0E-02	9.0E-02
Plutonium-239/240	~~**6.0E+00 µgm/gm**~~ 3.7E-2	9.6E-03
Total Alpha	~~1.0E-03~~	2.3E-01
	µgm/gm	µgm/ml
Al	3.3E+02	7.5E+01
Ba	6.0E+02	7.8E+01
Ca	1.8E+02	1.5E+02
Cd	1.1E+01	7.5E+00
Co	3.0E+00	3.0E+01
Cr	1.2E+02	1.5E+01
Cu	1.8E+01	1.7E+01
Fe	1.4E+02	1.5E+02
K	1.5E+03	7.5E+01
La	6.0E+01	3.5E+01
Mg	5.4E+02	1.5E+02
Mn	3.0E+02	1.5E+02
Mo	6.0E+00	9.0E+01
Na	1.5E+02	7.5E+01
Ni	1.6E+02	3.0E+01
Pb	6.0E+02	3.0E+02
Si	3.0E+03	1.7E+02
Sr	3.0E+02	8.7E+01
Ti	1.5E+02	1.7E+01
U	6.0E+02	6.0E+02
Zn	6.0E+00	1.65E+01
TOC	6.0E+01	1.5E+03
TIC	3.0E+01	1.5E+02
Cl	2.3E+02	3.0E+00
F	7.5E+03	1.5E+02
NO3	4.5E+02	3.0E+03
SO4	1.2E+03 (as S)	2.3E+03
PO4	6.0E+02 (as P)	2.5E+03

X 6.2 E-2 (for Pu-239)

10 times below level of concern

.37 uCi/g / 10 = 3.7×10-2/g

R.J Hallen via phone call with Mike Johnson 8:45AM 10/14

 and dry first before analytical.

Table 3. Description of Analyses

Constituent	Analysis Method	PNNL Procedure No.
Cs-137 (Eu-154, Eu-155)	GEA	PNL-ALO-450
Strontium-90 (Yttrium-90)	Separations and Beta Counting	PNL-ALO-476/431
Tc-99	ICP/MS	PNL-ALO-281
Am-241 (Cm-244, Pu-238, Pu-239/240)	Separation and AEA	PNL-ALO-417/496
Metal Ions (see Table 2 list)	ICP-AES	PNL-ALO-211/280
TOC/TIC	Hot Persulfate	PNL-ALO-381
Anions	IC	PNL-ALO-212
Hydroxide	Autotitration - EPA SW-846 Modified Method, 310(3)	PNL-ALO-228

Table 4. Physical Properties Measurements

Analysis
Bulk Density
Supernatant Density
Particle Size Distribution
Viscosity
Suspended Solids Loading:
- Weight % Insoluble Solids in Slurry

Test Instruction Supplement: Additional CUF Testing at 25% Dilution of Sr/TRU Precipitated Slurry

PNNL Test Instruction	**Document No.: BNFL-TI-29953-052** **Rev. No. 0, Supplement**
Title: Sr/TRU Precipitation of AN-107 Diluted Feed and Combined Removal of Entrained Solids and Sr/TRU Precipitate by Crossflow Filtration	
Work Location: RPL SFO HLRF	**Page** 1 **of** 8
Author: Richard T. Hallen	**Effective Date:** Upon Final Approval **Supercedes Date: New**
Use Category Identification: Information	
Identified Hazards: __Radiological __ Hazardous Materials __ Physical Hazards __ Hazardous Environment __ Other:	**Required Reviewers:** X Technical Reviewer ____ SFO Manager

Are One-Time Modifications Allowed to this Test Instruction? X Yes __ No

NOTE: If Yes, then modifications are not anticipated to impact safety. For documentation requirements of a modification see SBMS or the controlling Project QA Plan as appropriate.

On-The Job Training Required? ____ Yes or X No

FOR REVISIONS:
Is retraining to this procedure required? ____Yes X No

Does the OJT package associated with this procedure require revision to reflect procedure changes?
____Yes ____No X N/A

Approval	Signature	Date
Author	Richard T. Hallen	9/27/99
Technical Reviewer	RJ Brooks	9/27/99

4.0 Materials, Equipment, Supplies and Reagents Needed

4.1 Materials Required

1. Four 1 liter polyethylene bottles. They should be labeled as follows: "AN-107 DF CUF Filtrate," "AN-107 DF Filtrate (filter unit receiver)," "CUF Dewatered Slurry," and "CUF AN-107 DF First Rinse."
2. Four 100 mL bottles for each wash solution labeled wash 1 through 4. These can be filter unit bottles if small ones are available.
3. Two 10 liter containers, one labeled for the alkaline rinses and the other labeled for the acidic rinses.
4. Containers for draining from the bottom of the pump and from the sample valve.
5. 12 liters of 0.2 micron filtered DI water for determining clean water flux and for rinsing the CUF

4.2 Equipment

1. 1-L deadend filtration unit and four 150 mL units
2. 5-mL pipette (4 needed)
3. Four, 10 mL volumetric flask for density determination (ball flask)

4.3 Reagents Needed

1. 1 liters of 1M HNO_3 + ~0.1M Citric Acid
2. 1 liter of 5 ppm hypochlorite solution (pH > 7)
3. 250 mL of 0.01M NaOH (wash solution)

4.4 Other Supplies

1. Workplace Copy of Operating Procedure BNFL-TP-29953-020
2. Extra Copies of Data Sheets 1, 2, and 3
3. Laboratory Record Book
4. DAS disk for recording data

5.1 Pre-start for Sr/TRU Precipitation and CUF Operation

5.1.1 Inventory materials, equipment, supplies, and reagents to ensure all required items are available. Assure that all materials have been modified for remote handling.

5.1.2 Do the following and initial and date when each item is completed.

RJH Review PNNL Operating Procedure BNFL-TP-29953-020.

RJH Review the work instructions in BNFL-TI-29953-052. – supplemental

5.1.3 Conduct the "0.0 Pre-Start" operations in BNFL-TP-29953-020. Drain the system overflow container.

-/27/99 – CUF cleaned by external filter –

5.1.4 Perform "1.0 Start-Up" operations in BNFL-TP-29953-020 with 1 liter of filtered DI water with one variation: V4 the filtrate control valve should be closed. Run CUF for 5 minutes at between 4-6 gpm. In-line pressure should be varied from using V1 from 10 to 70 psig. Ensure that there are no leaks in the system. If leaks are detected, shut down system immediately.

5.1.5 Conduct the "10.0 Draining the system" operation in BNFL-TP-29953-020.

5.1.6 Perform "1.0 Start-Up" operations in BNFL-TP-29953-020 with 1.0 liter of filtered, distilled water with one variation: V4 the filtrate control valve should be closed. Run CUF for 5 minutes at between 4-6 gpm. In-line pressure should be varied from using V1 from 10 to 70 psig.

5.1.7 Conduct the "10.0 Draining the system" operation in BNFL-TP-29953-020.

5.1.8 Perform "1.0 Start-Up" operations in BNFL-TP-29953-020 with 1.0 liter of filtered, distilled water

5.1.9 Perform "6.0 Back pulsing" operations in BNFL-TP-29953-020.

5.1.10 Shut off the system and conduct the "10.0 Draining the system" operation in BNFL-TP-29953-020.

5.1.11 Perform "1.0 Start-Up" operations in BNFL-TP-29953-020 with 1.0 liter of filtered, distilled water.

5.1.12 **Determine clean water flux.** Conduct the "3.0 Operation during Ultrafilter Recycle Mode" operations in BNFL-TP-29953-020 using the conditions below. Filtrate flow rate should be monitored and data collected in the operating procedure. Each test should be performed for only 20 minutes and the system should be back pulsed. After each condition, the test engineer should initial and date the table below.

Condition	Flowrate (gpm)	Transmembrane Pressure (psig)	Initial and date when complete
1	4.20	10	RJ VA
2	4.20	20	RJ VA
3	4.20	30	NOT Run

see data sheet #1 - TI-052 Supp

5.1.13 Shut off the system and conduct the "10.0 Draining the system" operation in BNFL-TP-29953-020. Drain as much as possible water out of the system. Be sure to drain the pump also.

condition 1

M&TE List:

_____ Balance 1:

Calib ID ___________ Calib Exp Date__________

Location ___________

_____ Balance 2:

Calib ID ___________ Calib Exp Date__________

Location ___________

Thermocouple:

Calib ID ___________ Calib Exp Date_____________

Location ___________ Thermocouple type __________

Digital Thermometer:

Calib ID ____________ Calib Exp Date_____________

Location ____________

5.4.1 Verify that the M&TE List has been completed (and are working).

5.4.2 Conduct the "1.0 Start-Up" operations in BNFL-TP-29953-020. Transfer waste from bottles marked CUF Slurry and CUF Filtrate to slurry reservoir. Transfer the CUF Slurry first. There will likely be some solids left in the CUF Slurry bottle, try to minimize these by swirling the bottle during the transfer of the final small volumes of wastes. If significant solids remain, use the CUF Filtrate to wash the solids from the bottle. Record the weights of the bottles before and after transfer.

5.4.3 Record the weights of waste and bottles.

- DF-07 -08 -09 -10 added after tare

CUF Slurry and bottle 944.23 g
Empty bottle 106.65 g Weight transferred to CUF _________g

CUF Filtrate and bottle 1296.53 g
Empty bottle 106.64 g Weight transferred to CUF _________g

5.4.4 Record the level in the slurry reservoir sight glass.

Height NA inches

5.5 CUF Operation: Sr/TRU Precipitate Removal Test with AN-107 Diluted Feed

5.5.2 Conduct the "3.0 Operation during Ultrafilter Recycle Mode" operations in BNFL-TP-29953-020 using the conditions of 50 psid and 4.2 gpm. Filtrate flow rate should be monitored and data collected on a data sheet every 10 minutes over a period of 1 hour. Then back pulse twice and repeat the conditions and record data for 20 minutes. Then back pulse twice, open valve 1 to drop the pressure and turn off the pump.

5.5.2 Dilute the waste in the CUF by 25%. Use the 1st CUF Rinse solution. Shake well before transferring to the slurry reservoir. Approximately 450 grams should be used to dilute the slurry. Record weights below

1st CUF Rinse and bottle 1025.06 g
Remaining waste/bottle 618.55 g   Weight transferred to CUF 406.51 g

Record the level in the slurry reservoir sight glass.
Height ________ inches

5.5.1 Obtain slurry samples following "7.0 Slurry Sampling" in BNFL-TP-29953-020. The samples should be taken before any filtration tests. The first two slurry samples should not be saved, but dumped back into the slurry reservoir. Allow the waste to recirculate in the CUF for 5 minutes with no filtration, i.e. the CUF pump in operation, and the throttle valve completely open (no pressure or filtrate flux). While the waste is recirculating collect enough sample in a bottle to determine the slurry density.

Tare flask________
Flask plus 10 mL of filtrate________
Weight of 10 mL filtrate________   density of filtrate________g/mL

ND

Tare flask________
Flask plus 10 mL of filtrate________
Weight of 10 mL filtrate________   density of filtrate________g/mL

per Mike delete these samples/analyses

Then use ~~the slurry sampling~~ value to collect two 20-mL slurry samples in vials DF-12 and DF-13 for chemical analyses and two samples in DF-14 and DF-15 for physical property ~~measurements.~~ Use one for physical property measurements and store the other. Record the weight and sample number in Data Sheet 3.

5.5.2 Conduct the "3.0 Operation during Ultrafilter Recycle Mode" operations in BNFL-TP-29953-020 using the conditions below. Filtrate flow rate should be monitored and data collected as specified in the operating procedure. After each condition, the test engineer should initial and date the table below. If no filtrate flow occurs or the filtrate flow is less than 10 mL/min, discontinue testing, back pulse, and move to the next condition.

NOTE: Test conditions below are suggested and conditions do not need to be run for the full 1 hour if filtrate flux drops to low values. Conditions 4 and 5 should only be run if time allows. Skip and move on to condition 7. Condition 7 must be ran. Collect data at 10 minute intervals on data sheet.

could only reach 4.1 gpm

Condition	Flowrate (gpm)	Transmembrane Pressure (psig)	Initial and date when complete
1	4.20	50	RJH 9/30/99
2	4.20	30	RJH 9/30/99
~~3~~	~~4.20~~	~~70~~	delete
4	3.13	Optimum from 1-3 → 50 psig	
~~5~~	~~5.23~~	~~Optimum from 1-3~~	delete 9/30/99
6	4.20	50	RJH
7*	4.20	50	RJH 9/30/99

* back pulse after every 10 minutes of operation

Conditions 3+5 could not be obtained

5.5.3 Conduct dewatering in CUF. Follow procedure in BNFL-TP-29953-020. Conduct dewatering using condition 7. Collect the filtrate in the tared 1-L bottle labeled "AN-107 DF CUF Filtrate." Back pulse as necessary to keep filtrate flux to acceptable levels. The volume will only be reduced to ~800 mL in the CUF. Additional dewatering will be done using deadend filtration.

Tare weight of AN-107 DF CUF Filtrate Bottle 106.64
Bottle and filtrate ___________ Weight of CUF Filtrate_________

Determine the density of the CUF filtrate.

Do in SAL after composited filtrate

Tare flask____________
Flask plus 10 mL of filtrate_____________
Weight of 10 mL filtrate_________________ density of filtrate___________g/mL

Tare flask____________
Flask plus 10 mL of filtrate_____________
Weight of 10 mL filtrate_________________ density of filtrate___________g/mL

5.5.4 Obtain two 20-mL filtrate samples following "8.0 Filtrate Sampling" in BNFL-TP-29953-020 and using the pre-labeled sample vials after approximately half of the dewatering in the CUF has been completed. Record the weight and sample number in Data Sheet 3. One will be used for chemical and radiochemical analyses and one stored. – Sample composited filtrate in SAL

5.5.5 Conduct the "11.0 Shutting down" operation in BNFL-TP-29953-020.

5.6.1 Tare the 1 liter bottle labeled, "AN-107 DF CUF Dewatered Slurry."

Weight of bottle and lid ___106.65___ g reused original bottle

5.6.2 Conduct the "10.0 Draining the system" operation in BNFL-TP-29953-020. Collect slurry in 1 liter bottle. Make sure as much materials as possible has been collected. This material will be further dewatered using a deadend filter unit. Weigh bottle after all slurry has been removed from the CUF.

Weight of slurry, bottle and lid ____________________ g
Weight of material collected ____________________ g

NOTE: Proceed with rinsing the CUF and conduct 5.7 Dewatering using a Deadend Filtration Unit when time allows. This can start as soon as the initial rinse of the CUF is completed.

5.6.3 Conduct the "9.0 Rinsing the system" operation in BNFL-TP-29953-020. The first rinse should be done with 1 liter of distilled water. This liquid should be collected and saved in the container labeled "AN-107 DF CUF First Rinse." The second rinse should be done with 2 liters of filtered, distilled water, and the final rinse with 1 liter filtered, distilled water. The second and third rinses should be collected separately from the first in the alkaline rinse storage container.

NOTE: Once the CUF has been rinsed you may proceed to Section 5.7 and come back and complete the CUF cleanup later.

5.6.4 Perform "1.0 Start-Up" operations in BNFL-TP-29953-020 with 1.0 liter of filtered, distilled water.

5.6.5 Perform "6.0 Back pulsing" operations in BNFL-TP-29953-020.

5.6.6 Determine the clean water flux at 20 psid transmembrane pressure and flow rate of 4.2 GPM following "3.0 Operation during Ultrafilter Recycle Mode" operations in BNFL-TP-29953-020. Filtrate flow rate should be monitored and data collected in the operating procedure. Data should be collected for at least 20 minutes and the system should be back pulsed.

5.6.7 Shut off the system and conduct the "10.0 Draining the system" operation in BNFL-TP-29953-020.

5.6.8 Conduct the acid wash of the CUF unit with 1M HNO3/0.1-0.2M Citric Acid as described in the CUF operating procedure if needed to regain clean water flux. The acidic solution should be allowed to sit in the CUF overnight. When drained, the acidic solution should be placed in a separate container.

5.6.9 The CUF should be drained according to "10.0 Draining the system" operation in BNFL-TP-29953-020 and rinsed at least 3 times with filtered, distilled water to bring the pH back up to neutral. The acidic solutions should be placed in a separate container from the alkaline ones.

5.6.10 Perform "1.0 Start-Up" operations in BNFL-TP-29953-020 with 1.0 liter of filtered, distilled water.

5.6.11 Conduct the "3.0 Operation during Ultrafilter Recycle Mode" operations in BNFL-TP-29953-020 using the conditions below. Filtrate flow rate should be monitored and data collected in the operating procedure. Each test should be performed for only 20 minutes and the system should be back pulsed. After each condition, the test engineer should initial and date the table below.

Condition	Flowrate (gpm)	Transmembrane Pressure (psig)	Initial and date when complete
1	4.20	10	
2	4.20	20	
3	4.20	30	

5.6.12 Shut off the system and conduct the "10.0 Draining the system" operation in BNFL-TP-29953-020.

5.7 Dewatering with the Deadend Filter Unit

5.7.1 Tare the 1-L receiver bottle/lid and filter assembly/lid of the filtration unit.

Tare of receiver bottle ~~and lid~~ 131.05g lid 6.44g
Tare of the filter assembly and lid 104.30g

5.7.2 Assemble 1-L filtration unit and filter all of slurry in bottle labeled "CUF Dewatered Slurry." Solids should have settled on setting. Decant most of the supernate from the bottle and filter. The supernate should filter quite fast. When approximately half the material has been filtered, swirl the bottle to suspend the solids. Continue filtering the slurry. If excessive solids remain in the bottom of the bottle, small amounts of filtrate can be used to rinse the solids from the bottle. Record weight of empty bottle. Filter the solids until compacted on filter and no free liquid remains. Disassemble filter unit and weigh

Tare of receiver bottle/lid and filtrate ____________ Weight of filtrate ____________
Tare of the filter assembly/lid and wet solids ____________ Weight of wet solids ____________

5.7.3 The two 1-L bottles of filtrate, CUF filtrate and the deadend filtrate, should be transfer to SAL and combined to make a composite filtrate. Take duplicate samples of the filtrate composite. Record weight of each filtrate and vial used on Data Sheet 3.

Data Sheet 1: Operating Data

Sheet 1

Date: 9/20/99
Tank Number: AN-107 DF - Sr/TRU
Filter: Mott 0.1 µm Liquid service
Test Conditions: Low Solids
Operator: Ralph Lettau
Test Engineer: K Brooks

Test No.	Time	Chiller Temp.	Slurry Temp.	Slurry loop Flowrate	Filter Outlet Pressure	Permeate Pressure	Filter Inlet Pressure	Filtrate Flowrate Volume	Filtrate Flowrate Time	Filtrate Flowrate Flowrate	Tank Level	Comments
1	18:03	25.1	26.2	4.0	48	0	52	40	45.61			
1	18:04	23.6	26.8	4.1	48	0	50	20	46.85			
1	18:23	25.2	27.2	4.0	46	0	50	20	48.17			
1	18:33	27.1	27.5	4.0	47	0	51	20	49.09			
1	18:43	24.6	27.9	4.0	46	0	50	20	61.85			
1	18:53	22.9	28.2	4.0	46	0	50	20	65.35			
1	19:03	26.5	28.5	4.0	46	0	51	20	59.24			
	19:05	Started backpulse			19:12 Complete backpulse!							
		Performed second backpulse										
2	19:17	23.0	28.9	4.19	26	0	30	20	44.49			
2	19:27	22.2	29.1	4.2	27	0	30	20	58.70			
2	19:39	26.8	29.3	4.2	28	0	32	20	54.52			
2	19:47	26.0	29.4	4.2	29	0	33	20	59.49			
2	19:57	21.9	29.6	4.25	28	0	32.5	20	77.02			
2	20:07	23.7	29.7	4.2	28	0	31	20	72.74			
2	20:17	27.1	29.8	4.3	28	0	32.5	20	71.81			
	20:19	Started backpulse										
	20:27	Performed second backpulse										

Data Sheet 1: Operating Data

Sheet 2

Date: 9/20/99.
Tank Number: AN-107 – Sr/TRU
Filter: Mott 0.1 um Liquid Service
Test Conditions: Low solids
Operator: Ralph Lettau
Test Engineer: K Brooks

Test No.	Time	Chiller Temp.	Slurry Temp.	Slurry loop Flowrate	Filter Outlet Pressure	Permeate Pressure	Filter Inlet Pressure	Filtrate Flowrate Volume	Filtrate Flowrate Time	Filtrate Flowrate Flowrate	Tank Level	Comments
3	20:34	22.3	30.0	3.3	68	0	72	40	58.16			Running pressure booster
3	20:44	27.4	30.1	3.4	67.5	0	70	20	51.55			* All previous slurry temps
3	20:55	25.6	24.7	3.5	67	0	70	20	67.39			were taken of the viscometer
3	21:04	24.3	23.2	3.55	66±3	0	70±3	29	77.74			rather than the slurry tank!
3	21:15	24.7	24.3	3.4	67±3	0	71±3	20	77.21			We will have to use the chiller temp
3	21:24	27.2	26.8	3.45	67±3	0	71±3	29	80.38			
3	21:34	25.7	24.8	3.45	66±3	0	70±3	20	88.63			
	21:43	Completed backpulse 1										
4	21:50	21.7	20.4	3.05	48	0	50	20	38.15			Flow began high & dropped w/in first minute
4	22:01	25.2	24.9	3.05	~~48~~ 50	0	~~50~~ 51	20	61.15			
4	22:11	27.4	26.8	3.1	48	0	50	29	71.87			
4	22:23	25.6	25.0	3.15	49	0	51	20	85.86			
4	22:32	25.1	24.2	3.16	49	0	51	29	96.45			
4	22:46	24.6	24.1	3.15	48	0	51	20	101.63			
4	22:57	25.8	24.8	3.1	48	0	51	~~20~~ 10	54.60			
	Backpulsed twice											
5	23:15	22.6	22.2	4.3	47	0	51	20	38.06			Turned on pressure booster
5	23:26	27.7	27.4	4.6	46	0	50	20	51.56			P varies by ~5-6 psi
5	23:33	26.2	25.4	4.6	47	0	52	20	58.93			
5	23:43	23.8	22.5	4.5	47	0	52	20	66.70			
5	23:52	25.0	24.7	4.5	47	0	52	20	69.99			
5	00:07	26.2	25.3	4.5	47	0	52	29	76.02			
5	00:19	23.3	22.2	4.4	48	0	53	20	88.81			

– Back pulsed twice –

Data Sheet 1: Operating Data

Sheet 3

Date: 9/21/99
Tank Number: AN-107 DF -Sr/TRU
Filter: Mott 0.1 um liquid service
Test Conditions:
Operator: Don Rinehart
Test Engineer: Rich Hallen

Test No.	Time	Chiller Temp.	Slurry Temp.	Slurry loop Flowrate	Filter Outlet Pressure	Permeate Pressure	Filter Inlet Pressure	Filtrate Flowrate Volume	Filtrate Flowrate Time	Filtrate Flowrate Flowrate	Tank Level	Comments
6	00:40	25.7	24.6	3.97	47	0	51	20mL	42.01			
6	00:50	22.9	21.7	4.02	48	0	52	20	63.02			
6	01:00	25.5	25.6	4.15	47	0	51	20	67.99			
6	01:10	27.1	26.4	4.08	47	0	53	20	68.20			
6	01:20	21.8	23.1	4.05	47	0	51	20	85.81			
6	01:30	24.2	23.7	4.05	48	0	52	20	84.63			
6	01:35			vial#	DF-10							Sample collected
6	01:43	27.4	26.9	4.10	48	0	53	20	77.72			
— back pulse			twice									
Conditions for dewatering							50 psi and 4.0 gpm					chiller reset to 27.0 °C
	02:12	26.1	25.7	3.88	47	0	51	20	50.45			waste temp down to 20.8°
	02:24	27.9	27.3	4.08	48	0	53	20	61.03			reset to 26.0°C
	02:33	25.2	24.2	4.03	46	0	50	20	81.57			
— sample			DF-11									
	02:44		25.4	3.74	45		49					— maxed AIR OUT
	02:50		29.4	3.62	44		47		81.69			pressure & crossflow ↓
	03:15											
—		shut down CUF			and		drained					

Data Sheet 1

page 1 of TI-052 supplement

Date: 4/29/99

Tank Number: TI-052 supplement

Operator: Vaughn / Hooper

Test Engineer: Rich Hallen

Clean Water Flux

Test No.	Time	Chiller Temp.	Slurry Temp.	Slurry loop Flowrate	Filter Outlet Pressure	Permeate Pressure	Filter Inlet Pressure	Filtrate Flowrate Volume	Filtrate Flowrate Time	Filtrate Flowrate Flowrate (R meter reading)	Tank Level	Comments
Clean Water 1	17:31	21.2	20.3	4.3	8	0	12			5		
1	17:36	23.1	22.4	4.3	8	0	10			10		
1	17:41	24.6	24.2	4.3	8	0	10			12		
1	17:47	26.4	26.2	4.3	8	0	10			12		
2	17:49	27.0	26.8	3.8	18	0	20			25		
2	17:53	25.9	24.8	3.9	18	0	20			21		
2	17:57	23.9	22.2	3.9	18	0	20			18		
2	18:01	20.3	22.1	3.9	18	0	20			15		
2	18:04	21.4	19.8	3.9	18		20			15		
	Back pulse twice											
2-P	18:13	22.5	21.6	3.9	18	0	20			25		
2-P	18:18	24.5	24.0	3.9	18	0	20			17		
	Stopped – pressure pulsed with pump											
	external filter turned on – 18:22											
4/30/99 waste before dilution –					valve 4 opened		~~50~~	~~20~~	~~62.16~~	8:08		
	8:12	26.2	26.8	4.2	45	0	50	20	62.16			
	8:23	26.8	26.5	4.1	46	0	50	20	70.23			
	8:34	24.0	23.3	4.1	46	0	50	20	84.86			
	8:44	26.3	26.6	4.1	46	0	50	20	84.38			
	8:54	27.3	27.2	4.2	46	0	50	20	80.93			
	9:09	24.3	23.5	4.2	46	0	50	20	94.47			

Shut off

r of
TI-052
supplement

Data Sheet 1

Date: 9/30/99
Tank Number: AN-107 DF - TI-052 supplement - diluted to ~ 4.5M Na
Operator: Ralph Lettau
Test Engineer: Rich Hallen

Test No.	Time	Chiller Temp.	Slurry Temp.	Slurry loop Flowrate	Filter Outlet Pressure	Permeate Pressure	Filter Inlet Pressure	Filtrate Flowrate Volume	Filtrate Flowrate Time	Filtrate Flowrate Flowrate	Tank Level	Comments
—	Diluted w/ 1st CUF Rinse - recirculated - back pulsed twiced - slurry sampled											
1	9:45	26.8	27.2	4.0	47	0	51	30	55.59			Air at max.
1	9:52	27.2	27.1	4.1	47	0	49	30	71.09			
1	10:11	23.7	23.0	4.1	47	0	50	20	60.05			
1	10:24	27.2	27.6	4.1	47	0	50	20	56.07			
1	10:35	26.6	26.2	4.1	47	0	50	20	60.62			
1	10:46	23.9	23.2	4.1	47	0	50	20	68.35			
	- Back pulse twice - pressure 65 psig -											
2	10:58	24.3	23.9	4.2	26	0	30	20	59.73			
2	11:09	27.1	27.3	4.2	27	0	30	20	59.23			
2	11:22	24.8	23.9	4.2	26	0	30	20	70.81			
2	11:34	22.6	21.7	4.2	26	0	30	20	67.56			
2	11:46	26.0	26.0	4.2	26	0	30	20	70.67			
2	11:54	26.9	26.4	4.2	27	0	30	20	69.66			
2	12:08	22.6	21.4	4.1	27	0	30	20	84.39			
	- Back pulsed twice 8.83 seconds back pulse 7.68 second pulse #2											
4	12:23	24.8	24.7	3.1	50	0	52	20	42.06			
4	12:32	27.6	27.6	3.1	47	0	51	20	56.89			
4	12:44	24.0	23.1	3.1	47	0	50	20	73.31			
4	12:56	23.8	23.4	3.1	47	0	51	20	78.65			
4	13:05	26.8	27.1	3.1	48	0	51	20	75.38			
4	13:20	24.7	23.9	3.1	46	0	49	20	89.74			
4	[illegible]33	23.4	22.9	3.1	46	0	[illegible]	20	95.63			

Data Sheet 1

Date: 9/30/99
Tank Number: AN-107 DF T1-052 supplement
Operator: Ralph Lettan
Test Engineer: Rich Hallen

Test No.	Time	Chiller Temp.	Slurry Temp.	Slurry loop Flowrate	Filter Outlet Pressure	Permeate Pressure	Filter Inlet Pressure	Filtrate Flowrate Volume	Filtrate Flowrate Time	Filtrate Flowrate Flowrate	Tank Level	Comments
		Back Pulsed twice —					10.10 seconds			8.95 seconds		
6	13:48	27.9	28.5	4.1	47	0	51±2	20	39.69			
6	14:00	27.3	27.0	4.1	46	0	50	20	51.04			
6	14:10	24.8	24.0	4.06	47	0	50	20	61.15			
6	14:21	24.3	23.9	4.0	47	0	51	20	60.91			
6	14:31	27.4	27.7	4.17	47	0	51	20	59.43			
6	14:40	26.7	26.4	4.14	47	0	51	20	63.91			
6	14:51	24.8	24.2	4.11	46	0	50	20	72.16			
	15:02	Backpulsed twice - oil added to oiler										
7	15:11	26.4	26.8	4.10	47	0	50	20	45.17			
7	15:21	28.7	29.9	4.10	47	0	50	20	46.13			
	Back pulse — once											
7	15:26	27.3	27.0	4.16	47	0	50	20	41.91			
7	15:35	25.7	25.1	4.11	47	0	50	20	53.56			
	Back pulse - once											
7	15:40	24.1	23.3	4.07	47	0	50	20	47.18			
7	15:51	25.1	25.0	4.05	47	0	50	20	57.39			
	Back pulse - once											
7	15:58	26.6	26.5	4.08	47	0	50	20	45.02			
7	16:08	26.8	25.5	4.05	47	0	50	20	56.56			
	Back pulse — twice											

Page T of T1-052 supplement

Data Sheet 1

Date: 9/30/99
Tank Number: AN-107 DF
Operator: Don Rinehart
Test Engineer: Rich Hallen

50.50

Test No.	Time	Chiller Temp.	Slurry Temp.	Slurry loop Flowrate	Filter Outlet Pressure	Permeate Pressure	Filter Inlet Pressure	Filtrate Flowrate Volume	Filtrate Flowrate Time	Filtrate Flowrate Flowrate	Tank Level	Comments
7	16:20	23.9	23.4	4.15	47	0	51	20	~~26~~	~~end~~		
7	16:30	27.3	27.5	4.21	47	0	51	20	53.31			
	back pulse twice											
7	16:43	25.3	24.6	4.20	46	0	50	20	48.98			
7	16:55	24.2	23.4	4.2	47	0	51	20	61.46			
	back pulse — once											
DW	17:07	24.6	24.5	4.0	47	0	52	20	57.38			
	17:19	28.9	29.0	4.2	47	0	51	20	57.81			
	17:32	26.2	25.6	4.25	47	0	51	20	66.65			
	17:41 Bottle #1 CUF Filtrate removed											
	17:44 new bottle in place #2								~~77.16~~			
	17:45	24.3	23.4	4.2	47	0	52	20	77.16			
	18:04	26.6	26.3	3.48	43	0	47	20	89.52			
	18:29 filtrate collect stopped											

TI-052

Data Sheet 1: Operating Data

Clean Water Flux after AN-107 DF solids Removal and Clean up

Date: 10/05/99
Tank Number:
Filter:
Test Conditions: Clean Water Flux
Operator: Dave Ortiz
Test Engineer:

Test No.	Time	Chiller Temp.	Slurry Temp.	Slurry loop Flowrate	Filter Outlet Pressure	Permeate Pressure	Filter Inlet Pressure	Filtrate Flowrate Volume	Filtrate Flowrate Time	Filtrate Flowrate Flowrate	Tank Level	Comments
1	13:54	28.5	28.6	4.0	8	0	11					Rotameter = 13
1	14:04	23.7	22.0	4.1	9	0	12					Rotameter = 10
	14:11											Foamy in tank-added 600mL water
1	14:14	21.8	21.0	4.2	8.5	0	11.5					Rotameter = 8
	14:18	Backpulsed twice										
2	14:20	23.6	23.3	4.2	18	0	20					Rotameter = 30
2	14:22	24.3	24.1	4.2	18	0	20					Rotameter = 25
2	14:31	26.7	26.6	4.2	18	0	20					Rotameter = 21.5
2	14:40	23.5	22.7	4.2	18.5	0	20.5					Rotameter = 16
	14:45	Backpulsed twice										
3	14:45	21.9	20.8	4.2	28	0	30					Rotameter = 40
3	14:47											Rotameter = 31
3	~~14:56 FIB~~											
3	15:02	27.1	27.1	4.2	~~30~~ 29.5	0	32					Rotameter = 29
3	15:05	off	27.4	4.25	29.5	0	32					Rotameter = 29
3	15:08	off	28.6	4.2	29	0	31					Rotameter = 28

Prep of solutions for AN-107 Diluted feed
- TP-OB-rev.1 - T1-052 -

Need 1M Sr, 1M NaMnO4, and 3.5185M NaOH
and 1M HNO3 with 0.1-0.2M Citric Acid
for cleaning up CUF

makeup sheet for 1M Sr

Sr needs to be added to the diluted simulant for Sr/TRU decomtamination.
BNFL request 1M Sr(NO3)2

Balance number: 380-06-01-006

Balance ~~check~~ cal: 3-1-99

Balance ~~check~~ recal date: 2/00

D Hallen
9/13/99
T1-052
BNW-13733
page 44

Fischer Sci.
Makeup Sr(NO3)2 solution 1 M
use Sr(NO3)2 certified ACS
211.63 grams/mole (FW) (lot # 984987

Tare 200 mL volumetric flask 79.60 grams
add 42.326 grams Sr(NO3)2 flask + Sr(NO3)2 121.89 grams
actual weight of Sr(NO3)2 added 42.29 grams

add approximately 1/2 the volume of Milli-Q water and swirl until dissolved
fill to volumetric line with Milli-Q water. Record total weight: 311.30 grams ← for 200mL
calculate actual [Sr] = actual weight/211.63/volume in Liters = 0.99915 M
calculate density of solution, weight of solution/volume = 1.1585 grams/mL

$\frac{231.70}{200} =$

Transfer stock solution to poly bottle and label unique ID #

Transfer 142.8 mL to pour bottle 142.8 mL*density = grams to transfer 165.434 grams (396.914 target)
tare bottle: 231.48 g
add reagent and weigh: 397.19 grams
total weigh added: 165.71 g Total volume: 143.04 mL

Direction from Mike Johnson to use 0.01 M
NaOH for wash solution not DI water.

Project No. BNFL-Sr/TRU ppt 29953 Date of Work

Entered By D Hallen Date 9/13/99

Disclosed To and Understood By

Signed 1. Date

2. Date

Caustic needs to be added to diluted the waste before Sr/TRU decomtamination.
need 3.~~5~~185M ~~Sr(NO3)2~~ DMH ok STET NaOH

Balance number: 380-06-01-006 RDHaler 9/13/99
Balance check: Cal 3-1-99.
Balance check: 2/00.
recal date

Makeup NaOH solution 3.5185 M
use NaOH pellets 99.99% 02911MN - Semiconductor grade
40 grams/mole (FW) (lot # 06431MY
Tare 500 mL volumetric flask 188.20 grams +Stopper
add 70.37 grams NaOH flask + NaOH 258.47 grams
actual weight of NaOH added 70.27 grams

TI-052
BNW-13733
page 45

NaOH solution
Prep Sheet

add approximately 1/2 the volume of Milli-Q water and swirl until dissolved
fill to volumetric line with Milli-Q water. Record total weight: 749.51 grams 561.31/500
calculate actual [NaOH] = actual weight/40/volume in Liters = 3.5135 M
calculate density of solution, weight of solution/volume = 1.12262 grams/mL
Transfer stock solution to poly bottle and label unique ID #

Transfer 264.6 mL to pour bottle 264.6 mL*density = grams to transfer 297.045 grams
tare bottle: 350.95 and Lid (647.995 target)
add reagent and weigh: 648.31 grams
total weigh added: 297.36 g Total volume: 264.88 mL

Date prepared:
Prepared by:
Work Package Number:

d = 1.12262 g/mL

264.6

250 mL of 0.01 M NaOH for solids washing

X mL (3.5135 M) = 250 mL (0.01 M) = 0.71154 mL

.71154 mL $\times$ 1.12262 = 0.799 gram

250 mL flask
tare flask = 107.00 g
+ 3.5 M NaOH = 107.80 g
+ water 355.98 g
d = 0.9959 g/mL

transfer bottle
tare Bottle 350.62 g
+ NaOH 598.99 grams

Date of Work
Entered By RDHaler Date 9/13/99
Disclosed To and Understood By
Signed 1. Date
2. Date

Sodium permanganate needs to be added to the diluted simulant for Sr/TRU decomtamination.
BNFL request 1M $NaMnO_4$

NaMnO4 prep sheet →

Balance number: 380-06-01-006

Balance ~~check~~ cal dat: 3-1-99 .
Balance ~~check~~ recal date: 2/00 .

RD Hallen
9/13/99
TI-052
BNW-13733
page 46

Makeup $NaMnO_4$ solution 1 M
use $NaMnO_4.H_2O$
159.94 grams/mole (FW) (lot # A010675901
Tare 100 mL volumetric flask 67.62 grams
add 15.994 grams $NaMnO_4$ flask + $NaMnO_4$ 83.63 grams
actual weight of $NaMnO_4$ added 16.01 grams
add approximately 1/2 the volume of Milli-Q water and swirl until dissolved
fill to volumetric line with Milli-Q water. Record total weight: 177.05 grams
calculate actual [MnO_4] = actual weight/MW/volume in Liters = 1.0010 M
calculate density of solution, weight of solution/volume = 1.0943 grams/mL
Transfer stock solution to poly bottle and label unique ID #

Transfer 96.6 mL to pour bottle 96.6 mL*density = grams to transfer
105.709 g
tare bottle: 229.04g
(334.749g) target
add reagent and weigh: 334.88 g
total weigh added: 105.84 Total volume: 96.72 mL

Need 1 M HNO_3 with 0.1-to-0.2 M citric acid for cleaning CUF and Filter element after filtering Sr/TRU ppt.

Citric Acid · 1 H_2O – FW 210.14 Lot # 924580D Fischer

21.00 gram citric acid · 1 H_2O

769.23 gram 1 N HNO_3

approximately 3/4 full in 1-L bottle

Project No. ____________________ Date of Work ______
Entered By Richard D. Hallen Date 9/14/99
Disclosed To and Understood By
Signed 1. ____________________ Date ______
2. ____________________ Date ______

Calibration of graduated filtrate collection/flux measurement glass piece of CUF - with valve 6 in open position - i.e. side arm also fills with filtrate. Use autopipetter 237RA1-10-01-431920

pipette check

10.00 mL = 10.0447 g = 10.0272 g = 10.0390 g

bottom outlet plugged - Valve 6 opened - filled to 10 mL mark on graduated cylinder - some drops remain on sides of cylinder.

add	record volume reading
10 mL	18.6 mL
10 mL	27.0 mL
10 mL	35.7 mL
10 mL	44.2 mL

(only graduated to mL estimate the tenth of mL)

10 mL = 8.6 mL 10 mL = 8.4 mL 10 mL = 8.7 mL

10 mL = 8.5 mL

Calibrate of CUF flow measurement cylinder with valve 6 open
(the side arm also fills so readings need adjusted)
cylinder filled to 10 mL to start

volume added	reading	reading change
10	18.6	8.6
10	27	8.4
10	35.7	8.7
10	44.2	8.5
		8.55 0.129099
	average	stdev

calculation of AVE + stdev

KJ Haller 9/14/99 BNW-13733 page 47

so a reading change of 0.855+/-0.0129 mL is 1 mL actual volume of filtrate
correct filtrate flux data based on reading to actual volume by dividing by 0.855

Project No. ______ Date of Work ______

Entered By KJ Haller Date 9/14/99

Disclosed To and Understood By

Signed 1. ______ Date ______

2. ______ Date ______

12:00 AM

9/20/99 AN-107 DF Sr/TRU PPT and CUF

Stir Bar 38.4144 grams

Balance 384-06-01-004
8/2000

Vial#	Tare	
DF-01	~~16.9127g~~	17.0230g
DF-02	16.8683g	
DF-03	16.9718g	
DF-04	16.9215g	
DF-05	16.8945g	
DF-06	16.9470g	
DF-07	16.9241g	
DF-08	16.8017g	

vial # RJH

Lids on Jars were Stuck

Bottle UFA had ~ 1/2 inch settled solid that were not easily transferred. Waste from UFB was ad to UFA and well mixed. All other bottles were stille Shaken well before transfer.

UFB had a very small amount of solid that didn't transfer.

Bottle UF~~Q~~D Lid stuck - 2:12:40 - use lid remover
RJH

Bottle UFE - had some chunky solids - ~1750 mL

Caustic Added Start 2:32 Temp 32.2
stirred until

1M Sr added 3:29 3:40 addition completed
at 3:35 scan started 32.9°C

Project No. ______ Date of Work ______

Entered By Richard D. Hale Date 9/20/99

Disclosed To and Understood By

Signed 1. ______ Date ______

2. ______ Date ______

1M NaMnO4 added 3:46 temp 32.8°C
finished at 3:57 about 2000 mL

4:38 ~50 mL removed for samples + Density measurement

4:45 heater turned on

reached ~45°C at 5:00

used 20 mL syringe
2-3 mL filter then plugged
filter changed plugged again 2-3 mL
new filter only a mL or so
used 4th filter - plugged -

vial + waste
DF-01 - 27.743 g filtrate

DF-02 - 40.485 g

Sample DF-02 not filtered!

6:00 temp 49.4°C

6:40 temp 49.0°C 7:20 48.7°C 8:30 48.4°C

8:55 48.3°C 9:10 48.0°C heat turned off

SKY * CUF had last of DI rinse H_2O as lay-up Solution..

Turned on the CUF to recheck H_2O flux:
Tank was foamy white. When V4 was opened
Rotometer reading went to ~10. Then quickly
dropped off. Went down to R=0 with-in 5 min.
Backpulsed twice - (second backpulse the chamber filled
much more quickly) then drained completely.

10:00am Refilled with 1L clean DI H_2O - ran T=0 R=20
System must be cleaned more before slurry addition. T=6 R=~8

10:50 samples of digested slurry taken

stirrer unplugged?? DF-03 42.068 g DF-04 41.622 g

Project No.________________ Date of Work________

Entered By Deborah T. Hallen Date 4/20/99

Disclosed To and Understood By

Signed 1.________________ Date________

2.________________ Date________

 9/20/99

TJB 9/20/99

12:47 pm 0.05 μ Cartridge filter hooked up and running.

ALA

Backpulsed in 2 chambers of clean DI from top of system. Added the remainder of a L H_2O to the tank before by-pass to Cartridge filter was opened.

1:22 V10 closed (filter by-pass valve)
V4 open P = ~20 F = ~3.8 gpm

T = 0 R = ~35
T = 8 R = ~~38~~ 36 ALA 9/20/99
T = 20 R = 33

System clean enough to run.

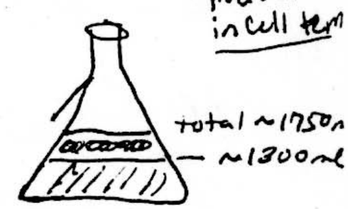
1:55 empting system

* dropped Flow to ~ 1.2 gpm 10 psig before dumping.

2:54 * Note T of slurry is still reading 34.7. So took the thermocouple out to see what it is reading in air ~~s~~ still 34.4 C - (some jumping in the 10th's of a degree)

∴ Thermocouple may not be reading correctly. → Talked to Mac
ALA * 4:50 after Thermocouple had been removed for ~5 min T ≅ 32 C & He said that is prob accurate in cell temp
Using a magnet to remove the stir bar

Visual before disrupting the settling
The slurry has settled into two visible layers. The bottom layer is a greenish brown and the top is a dark Navy blue/purple almost black.



Project No. [signature] Date of Work 9/20/99
Entered By [signature] Date 9/20/99
Disclosed To and Understood By
Signed 1. Richard T. Hallen Date 10/25/99
2. Date

3:11 Stir bar removed.

3:40 wt of vessel with slurry 3609.54g

3:59 AN-107 in and CUF on -
F ≅ 1.0 gpm Range (.92 - 1.1)
P = 0 psig - ~~CUF VI~~ (V1 open completely)

4:08 Visual check shows slurry not mixing well. Increased flow rate to 1.8 gpm - mixing slow but well.

4:13 Taking 1st Rheology sample.

4:50 * At about 4:00 Thermocouple lead that was in the slurry ~~Set~~ was unplugged and used to monitor the Rheology water bath.

17:45 Completed 2nd rheology sample

17:51 DF-05 taken as slurry sample Wt: 41.677g

17:53 DF-06 taken as slurry sample Wt: 41.697g

18:03 Started test w/ Condition #1 required air booster on 65 psi

18:21 Pulled filtrate sample DF-07 Wt = 38.033g
Heat Exchanger is leaking slightly.

Project No. ______ Date of Work ______

Entered By [signature] Date 10/25/99

Disclosed To and Understood By

Signed 1. [signature] Date 10/25/99

2. ______ Date ______

~~Tare Wt~~	Sample #
16.9849	DF-09
16.9114	DF-10
16.9553	DF-11
16.9620	DF-12
16.8648	DF-13
16.9018	DF-14
16.9472	DF-15

20:50 Discovered that TC was still plugged into viscometer! Chang

21:01 Took sample of filtrate during condition #3.
DF-08 35.385 g

22:19 Checked chiller liquid level. Found to be ~1" from the top.

~~22:55~~ JB
0:04 Filled DF-09 during test #5 Mass = 35.638 g

AN-107 Deadend Filtrate Bottle (w/ lid) 136.45 g
AN-107 DF Filter + Lid 104.78 g
> Bottle was banded - tare weight 198.66 g wb

at the end of condition 6 filtrate sample collected - DF-10 - 35.537 grams

1/2 way through ~~B~~ dewatering ~400 mL collected - DF-11 - 34.211 grams

CUF drained - small amount of slurry spilled
very thick slurry

At 04:56 the deadend end filter vacuum was turned on.

At 06:38 filter contains 6 ~~500~~ mL - ~80 ~~100~~ mL's filtrate in reserv

Project No. ______ Date of Work ______

Entered By [signature] Date 9/21/97

Disclosed To and Understood By

Signed 1. ______ Date ______

2. ______ Date ______

Cuf rinsed w/ 1 liter of water
1st rinse collected in 1 liter bottle
2nd rinse water added = 1 Liter } collected in
3rd rinse water added = 1 liter } 2 liter bottle

1 liter of water added to CUF

test condition 20 psi + 4.2 gpm

Clock	PSI	Time	volume	Temp	GPM	R-~~~
8:12	20	14.62	340 mL	+30	4.1	~4
8:31	20	29.96	30		4.0	
8:39	20	30.24	30 mL	16.8		1

CUF not cleaned up with water alone.
Drain and wash with acid
At 10:06 Deadend filter at 500 mL
Acid solution recirculated in CUF
flow at 20 psi = 20   Flow ≅ 4.2   R ≅ 10
10:27 - after back pulse w/ acid   -2- 23 (20 psi)
10:51 - Deadend filter @ 475 ml
10:55 - Backpulsed 2 times   R = 25

Project No.__________ Date of Work__________
Entered By [signature] Date 9/21/99
Disclosed To and Understood By
Signed 1.__________ Date__________
2.__________ Date__________

9/21/99

11:09 R ≅ 22 (Still 20 psig ≃ 4.2 gpm)

11:15 R ≅ 22.5 - Doing 10 back pulses

11:24 after back pulses R ≅ 28
Shutting down for soak ~1 hr.

~1:15 - Back on running R = 40 20 psig ~ 4.0 gpm

1:55 Decanting liquid level ~420 ml - dead end filter

1:55 R = 35 ~20 psig ~ 3.8 gpm

2:00 - Turned on chiller (Tacid = 47.1 C)
Back pulsing 10 times

Visual of inside of tank - Acid is white (entrained air) &
mixing well

2:35 R = 32 20 psig
Back pulsing 2 times

2:40 Draining Acid (Complete drain)

3:11 1st rinse in and running R = 40
1st rinse was 1.5 L DI H_2O. Backpulsed
entire amount through top to wash filter.

3:33 Decanting liquid level (An-107) ~375 ml - dead end filter

3:43 2nd wash in and running - 1 L DI H_2O
R = 35 P = 20 F = ~4.0 gpm

3:49 R = 30

4:40 Draining 2nd Rinse.

4:55 3rd rinse in and running (2 bambers entered from top)

5:08 Draining 3rd rinse

5:35 4th Rinse in and running - (most entered back pulse though the top)

Project No. ____ Date of Work 9/21/99

Entered By [signature] Date 9/21/99

Disclosed To and Understood By

Signed 1. [signature] Date 10/25/99

2. ____ Date ____

6:00 Turned off vacuum on ~~Decanting~~ Deadend / filtration system.

6:41 pH of 4th rinse is ~4.0 on pH paper.
Adding another 3/4 L of H_2O

Starting a water test

T=0 R=22.5
T=5 R=18.5

Not clean yet. –

9/22/99 filter Top tare with Lid 104.78g
BNFL has instructed us to recombine solids and filtrate for additional CUF testing

the filter assembly had ~350mL in it – 100mL of settled solids and 150mL supernate ~400 mL of filtrate had been collected the filtrate wash used to wash solids out of filter. Solid were very compacted. A pipette was used to mix filtrate and solids. After 4 – ~100 mL washes, solids still remain in filter. Weight of filter bottom with band = 200.31 g. CUF Slurry bottle w/slurry = 914.61 filter + Lid = 161.75g ⇒ so about 56.97 gram solids remain in filter

Project No. ______ Date of Work ______
Entered By [signature] Date 9/23/99
Disclosed To and Understood By
Signed 1. ______ Date ______
2. ______ Date ______

9/24/99 - solids were transferred from filter to CUF Slurry Bottle

filter + solids 321.94 g

filter only 292.22 g

29.72 gr remained in filter is probe half the

filter + cap + 106.38 g

1.45 grams left in filter

new tare clean 104.78 g

*CUF Slurry total weight 944.23 g

CUF Filtrate 1296.53 g

CUF 1st Wash 1025.06 g

*Caution CUF Slurry Bottle has a disposable pipette in it that will need to be removed.

9/29/99 CUF moved into position 1:37 - external filter hooked up to clean up CUF - 1:48 recycled water at 15 p and 1.9 gpm. Turned chiller on.

Project No. ______ Date of Work ______

Entered By [signature] Date 9/29/99

Disclosed To and Understood By

Signed 1. ______ Date ______

2. ______ Date ______

Estimation of Volume

clock sync.

my watch 2:23

camera ~~WClock~~ 2:29:27

646 of slurry

~~202~~923.65 mL filtrate

1569.6 mL - 25% dilution

estimate 1.96 L total

~ 400 mL (g) of 1st wash

recover filtrate from samples - DF-07, DF-08, DF-09, and DF-10

~ 80 mL more filtrate

valve 4 opened

flowrate 22 at t = 0 4.2 gpm 20 psi inlet

R = 18 at t = 5 min 4.3 gpm 20 psi

Back pulsed twice - then recycle through filter again. 3:00

Chiller - temp control sensor came unplugged.

15:52 - R = 26 at 20 psi and 4.2 gpm

backpulsed twice 15:54 R = 26 t = 19.6 °C

16:09 R = 21 t = 23.3 °C

16:14 R = 21 t = 25.1 °C

16:24 R = 19 t = 25.0 °C

16:44 clean water back pulse in system twice and external filter turned back on.

18:22 turned on recycle to external filter

				R=
18:56	20 psi +	4.3 gpm	21.8 °C	25
19:01	20 psi +	4.3 gpm	24.1 °C	21
19:08	20 psi		25.6	18

Project No. ______ Date of Work ______

Entered By [signature] Date 4/29/99

Disclosed To and Understood By

Signed 1. ______ Date ______

2. ______ Date ______

— Back pulsed twice —

19:15 20psi + 4.0gpm 23.9°C R meter 26

19:18 20psi + 4.0gpm 22.5°C 20

7:15 9/30/99 Drain Water out of CUF Cell temp 29.7°
(CUF slurry had DF-07 DF-08 DF-09 + DF-10 ac

add CUF Slurry — Slurry bottle rinsed out with filt-

add CUF filtrate —

Valve 1 fully open recirculate waste at ~2 gpm for 10 minutes 7:49

7:59 — target 50 psi + 4.2 gpm

Vial weights DF-13 16.8617g for dilute slurry sampl

DF-14 16.8970g — physical prop measurement

tareweight AN-107 CUF Filtrate #2 104.42

— 1st wash solution added approximately 1/2 of bottle

9:23 back pulse twice

9:32 — start slurry sampling

1st sample DF-13 — filled to ~20 mL — 2 valve sequences

DF-13 — 39.977g —

2nd sample DF-14 = 40.473g —

9:43 started condition #1

CUF Filtrate Bottle empty = 106.64g

CUF Dewatered Slurry empty = 106.65g

CUF 1st Rinse 1/2 full = 618.55g

Project No.________ Date of Work________

Entered By [signature] Date 9/30/99

Disclosed To and Understood By

Signed 1.________ Date________

2.________ Date________

- CUF pump at max and only get 4.1 gpm at 50 psi
So re-evaluate matrix - condition 3 + 5 not run

CUF Filtrate #2 831.23g

CUF filtrate (#1) 936.62

CUF Dewatered Slurry 941.31g

band added to filter bottom - CUF Deadend filter
reweighed with Top + cap 296.79g

19:13 filter Vacuum turned on volume ~ 625mL

empty CUF Dewatered Slurry 110.44g

500mL of water added to CUF for 1st Rinse -
collected in bottle labeled CUF 1st Rinse

20:02 filter volume at 575mL ~1mL/min

20:15 Rinse #2 complete - 1 Liter

20:30 Rinse #3 complete - 1 Liter

20:37 filter volume at 550mL

20:42 4th wash R=20 20psi 4.2gpm

Clean water flux

20:43 30mL in 19.95 seconds 22.3°C

20:48 20mL in 32.73 seconds 22.8°C

CUF Shut down 8:59
Vacuum turned off

Project No. ______ Date of Work ______

Entered By [signature] Date 9/30/99

Disclosed To and Understood By

Signed 1. ______ Date ______

2. ______ Date ______

10/1/99 8:00 - Vacuum turned back on
11:25 - 425mL in filter
3:13 350mL in filter
75mL in 4 hours - have another 250mL to ra
- So ~ another 12 hours -

10/4/99 - vacuum off over weekend and sampled filter

-11:36 acid placed in CUF

- AN-107 DF Dead end filtrate - Bottle w/ camp and cap - with filtrat
907.44 grams

filter assembly + new bottle + band + wet solids
395.92 grams

estimated 80 mL of solids

0.01M NaOH transferred to empty 3.5M Bottle (triple rinsed

tare of wash solution Bottle 583.30 grams

12:07 first wash started 459.29 grams after 1st wash removed; Bottl
filter very fast - went right through

Project No. ______ Date of Work ______
Entered By ______ Date 10/4/99
Disclosed To and Understood By
Signed 1. ______ Date ______
2. ______ Date ______

- 1st wash collected

12:19 CUF valve 4 open and acid run through ~~the~~ filter at 12 psi R = 25

13:45 backpulsed w/acid twice then at 18 psi R = 30

tare 706.17g more 0.01M added to NaOH Bottle for solids washing

tare of wash solution bottles

1st wash AN-107DF	tare	230.33 grams
	+washsolution	350.86 grams
2nd wash AN-107DF	tare	229.99 g
	+wash solution	404.77 grams
3rd wash AN-107DF	tare	231.74 g
	+wash solution	370.18 g
4th wash AN-107DF	tare	228.42 g
		415.63 g

Filter Bottom + first wash solution 301.11 grams

tare 180.43 grams bottom after 1st wash poured out

14:34 second wash solution added - tare 0.01M Bottle 529.07 gr

Vacuum Not turned on - Solid swirled/washed

Project No. ______ Date of Work ______

Entered By [signature] Date 10/4/99

Disclosed To and Understood By

Signed 1. ______ Date ______

2. ______ Date ______

14:47 filter backpulse twice with acid

t=0 15 psi R=30 t=10 min 15 psi R=25

20 psi = R=33

15:06 backpulsed twice t=0 20 psi R=35

pressure increased to 30 psi R=55

acid drained + water added - 1 Liter

wash solution filtered overnight w/o vacuum

10/05/99 355.59 gram filter bottom + second wash

2nd wash solution + bottle 404.77 grams

Wash solution and bottle 528.94 grams

8:35 3rd wash removed 390.92 grams - wash solution/bottle

3rd wash added vacuum not turned on

8:45 CUF started with 1st acid rinse backpulsed 3 times then drained

An additional 100 ml wash solution added to bottle 525.61 g

9:25 Clean water added - backpulsed 3 times

flux at 20 psi and 4.0 gpm = 32 = R

drained

9:44 new/clean water 20 psi + 4.0 gpm R=32

backpulsed 3 times 20 psi + 4.0 gpm R=35

Project No. ______________ Date of Work ______________

Entered By Richard L. Hallen Date 10/5/99

Disclosed To and Understood By

Signed 1. ______________ Date ______________

2. ______________ Date ______________

psi = 30 crossflow = 4.0 R = 55
psi = 10 crossflow = 4.3 R = 15
pH 3.5-4.0 – so one more rinse
CUF drained + ~~so~~ clean water added

10:13 psi = 20 3.9 gpm R = 32

10:16 3rd wash completed – NO vacuum was used
washed filter bottom 320.72 g a few drop ran down outside of bottle bottle + 3rd wash = 320.18 g
4th wash added – 12:50 vacuum turn on to dry out solids
13:18 vacuum turned off
Wash bottle empty – 351.41 gram Added 4th wash = 174.28 gram

	0.01M NaOH used	filtrate collected	amount of wash solution sample
1st wash	124.01 g	120.68 g	120.53 g
2nd wash	177.10 g	175.16 g	174.78 g
3rd wash	138.02 g	140.29 g	138.44 g
4th wash	174.28 g	187.41 g	187.21 g

filter bottom + 4th wash = 367.76 grams 4th wash + bottle = 415.63 grams
filter bottom empty = 180.35 grams
AN-107 DF SOLIDS – tare bottle empty 258.17 grams
– 90.90 grams wet solids bottle/jar + solids 349.07 grams
filter top/cap + wet solids 201.85 grams

filter top/cap with solids removed 109.68 grams

14:00 Performing clean water flux with CUF
10/06/99 external filter placed in line to cleanup system

8:42 set at 20 psi + 2.0 gpm 11:43 backpulsed 3 times
flow 4.2 gpm psi = 20 R = 25 t = 3 dropped to R = 20

Project No.______ Date of Work______
Entered By RJ Hallen Date 10/6/99
Disclosed To and Understood By
Signed 1.______ Date______
2.______ Date______

Analytical Samples to transfer to SAL

Physical ~~Measurement~~ Samples for Paul Bredt:

DF-05, DF-06, and DF-13

Samples for Chemical Analyses

DF-01, DF-02, DF-03, DF-04, DF-11, DF-14

All ready in SAL are DF-20 and DF-21 (filtrate composite

Composited samples SAL #4 "Labeled"

		inlet			slurry
13:53	back pulsed 3 times	psi = 20	4.5gpm	R = 28	temp = 2[illegible]
	t = 10min	psi = 20	4.3gpm	R = 26	temp = 23.7°C
	t = 20min	psi = 20	4.3gpm	R = 20	temp = 19.9°C
	outlet pressure = 18psi				
	t = 30min	psi = 20	4.2gpm	R = 18	temp = 25.5

Acid for CUF cleaning = 2nd cleaning citric acid hydrate 24.40 gram
add 1024.05 grams 1M HNO_3

0.01M NaOH wash solution used for 2nd-on washes 1.43mL (1.60 grams) of 3.5 M NaOH added 499.94 grams H_2O.

10/13/99 Sulfate Removal with Liu Resin

approximately 2 grams of sulfate selective resin was provided to BNFL for AN-107 archive testing - resin prepared by Jun Liu - PNNL

Project No. ______ Date of Work ______

Entered By [signature] Date 10/13/99

Disclosed To and Understood By

Signed 1. ______ Date ______

2. ______ Date ______

Additional Calculation for report — AN-107 CF

for Anion data we need to account for the nitrate added with the $Sr(NO_3)_2$ — so this is 2 X .075M for the original solution which was further diluted by 19% for the additional CUF testing

DF-20 + DF-21 Nitrate AVE = 87400 ug/g
density = 1.2414 g/mL MW NO_3^- = 62 g/mole

$$\frac{.15\,moles}{L}\left(\frac{62g}{mole}\right)\left(\frac{mL}{1.2414\,g}\right)\left(\frac{L}{1000\,mL}\right) = .0075\ g/g \text{ or } 7500\ ug/g$$

$$\frac{7500\ ug/g}{\text{Mass Dilution} = 1.19} = 6295\ ug/g$$ of Nitrate in final solution is from added $Sr(NO_3)$

the added Sr is going to ppt CO_3^{2-} = 1:1 ratio
$CO_3^{2-} \Rightarrow TIC$

$$\frac{0.075\,moles}{L}\left(\frac{12g}{mole}\right)\left(\frac{mL}{1.2414g}\right)\left(\frac{L}{1000mL}\right) = 7E^{-4}\ g/g \text{ or } 725\ ug/g$$

$$\frac{725\ ug/g}{\text{Mass Dilution} = 1.19} = 609\ ug/g$$ removed from final filtrate

Calculate the expected yield of solids from Sr/TRU removal treatment

Project No. ______ Date of Work ______

Entered By [signature] Date 2/11/2000

Disclosed To and Understood By

Signed 1. ______ Date ______

2. ______ Date ______

total weight of initial waste treated → 1845.438 grams

We added 165.367 grams of 0.999 M $Sr(NO_3)_2$
(d = 1.1585 g/mL) = 0.1426 moles Sr added
= 12.49 grams Sr added

We added 105.558 grams of 1.001 M $NaMnO_4$
(d = 1.0943 g/mL) = 0.0966 moles ~~$~~ Mn added
=

for reagents addition only

Sr ppts as $SrCO_3$ = 0.1426 moles $SrCO_3$ = 21.05 grams
FW $SrCO_3$ = 147.63 g/mole

Mn ppts as MnO_2 = 0.0966 moles MnO_2 = 8.39 grams
FW MnO_2 = 86.94 g/mole

To calculate how much ppts came from soluble species – take initial concentration [ ] ug/g (1845.438) g × % removed / 100 = ugrams removed

	element	grams	likely species	(FW)	grams ppt
40.08	Ca	0.207	$CaCO_3$ (CaF_2)	100.09	+ 0.517 grams
51.996	Cr	0.148	$Cr(OH)_3$	102.999	0.2932 grams
55.847	Fe	2.280	$Fe(OH)_3$	106.85	4.362 grams
54.938	Mn	0.600	MnO_2	86.94	+ 0.950 grams
207.2	Pb	0.245	PbO/OH/CO_3?	223.1994	0.2634 grams
87.62 (MW)	Sr	− 0.314	removed $Sr(CO_3)$	147.63	− 0.529 grams

Project No. ______ Date of Work ______
Entered By [signature] Date 2/11/2000
Disclosed To and Understood By
Signed 1. ______ Date ______
2. ______ Date ______

Sr/TRU ppt Solids Composition/yield

Component	As	Reagent	+ Solution	= total
Sr	$SrCO_3$	21.05	−0.529	20.521
Mn	MnO_2	8.39	+0.950	9.340
Fe	$Fe(OH)_3$	~~4.3620~~ 0	4.362	4.362
Ca	$CaCO_3$	0	0.517	0.517
Cr	$Cr(OH)_3$	0	0.2932	0.293
Pb	PbO	0	0.2639	0.264

predict that the ppt is **35.297 grams**

Paul report undissolved solids content of
0.62% X 1845.438 grams = 11.44 grams

PPT

gram		As ICP element	g/g	ug/g
20.521	$SrCO_3$	12.1794	0.3451	345000
9.340	MnO_2	5.902	0.1672	167000
4.362	$Fe(OH)_3$	2.28	0.0646	64600
0.517	$CaCO_3$	0.207		
0.293	$Cr(OH)_3$	0.148		
0.264	PbO	0.245		

Sr predicted to be 345000 but only 272500 —
So have $\frac{35}{X+35} = 0.78973 = 9.3189$ grams of solids

Project No. ______ Date of Work ______
Entered By [signature] Date 2/11/2000
Disclosed To and Understood By
Signed 1. ______ Date ______
2. ______ Date ______

Results suggest 9.3189 grams of entrained solids —
major components of entrained solids
are Na, Al, Si, Fe, and Mn.

Combining we get 44.616 grams of solid
in a total of 1845.438g waste
296.548g 3.51 M NaOH
165.367g 1.0M $Sr(NO_3)_2$
105.558g 1.0 M $NaMnO_4$

2412.911 grams

1.85 wt% Solids

FW 88 Oxalate = 23300 µg/g = "6350" as TOC

If we take 0.62% + Sr/TRU ppt. = 1.94 wt%
(1.937 wt%)

Project No. ____ Date of Work ____
Entered By [signature] Date 2/11/2000
Disclosed To and Understood By
Signed 1. ____ Date ____
2. ____ Date ____

APPENDIX B: ANALYTICAL DATA

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Radioanalytical Applications Team

11/15/1999

Client : Hallen

Cognizant Scientist: LR Greenwood   Date : 11/15/99

Concur : T Trang-le   Date : 11/15/99

Procedure: PNL-ALO-450, 476, 4001, 417, 496

Measured Activities (uCi/g) with 1-sigma error

ALO ID. Client ID	Alpha Error %	Sr-90 Error %	Pu-239 + Pu-240 Error %	Pu-238 Error %	Am-241 Error %	Cm-243+ Cm-244 Error %	Cm-242 Error %
PB-0070 Process Blank	<6.E-5	7.39E-4 11%	2.20E-6 14%	2.72E-6 13%	3.25E-6 14%	3.35E-6 13%	<2.E-7
00-0070 DF-01		9.46E-1 3%			1.01E-2 4%	4.24E-4 6%	3.86E-5 15%
00-0070 Rep DF-01		1.04E+0 3%					
RPD		9%					
00-0071 DF-11		5.85E-1 3%			9.36E-3 4%	3.89E-4 5%	4.17E-5 10%
00-0072 DF-20	8.10E-3 3%	4.69E-1 3%	6.54E-4 4%	1.72E-4 6%	5.11E-3 4%	2.47E-4 5%	2.84E-5 11%
00-0072 DUP DF-20	8.07E-3 3%	4.78E-1 3%	6.41E-4 4%	1.72E-4 5%	4.83E-3 4%	1.82E-4 5%	1.71E-5 12%
RPD	0%	2%	2%	0%	6%	30%	50%
00-0072 Rep DF-20	8.26E-3 3%		6.43E-4 4%	1.61E-4 5%	4.74E-3 4%	2.32E-4 5%	2.08E-5 13%
00-0073 DF-21	7.72E-3 3%	4.81E-1 3%	6.18E-4 4%	1.66E-4 6%	5.04E-3 4%	2.43E-4 5%	2.42E-5 12%

RT HALLEN
Date 11/19/99
~~Route~~
File TI-052
Copy record

Measured Activities (uCi/g) with 1-sigma error

ALO ID Client ID	Alpha Error %	Sr-90 Error %	Pu-239 + Pu-240 Error %	Pu-238 Error %	Am-241 Error %	Cm-243+ Cm-244 Error %	Cm-242 Error %
00-0074 Wash Composite	6.95E-4 12%	9.96E-2 4%	2.46E-5 4%	7.46E-6 7%	2.96E-4 4%	1.83E-5 6%	1.74E-6 16%
00-0074 DUP Wash Composite	6.39E-4 13%	9.62E-2 4%	2.41E-5 4%	6.48E-6 7%	2.82E-4 4%	1.40E-5 9%	1.03E-6 28%
RPD	8%	3%	2%	14%	5%	27%	51%
00-0079 DF-02		4.12E-1 3%			4.82E-3 4%	2.10E-4 6%	2.42E-5 15%
00-0080 DF-03		1.95E-1 3%			4.03E-3 4%	1.79E-4 6%	1.89E-5 15%
00-0081 DF-04		3.94E-1 3%			4.64E-3 5%	2.00E-4 8%	2.22E-5 20%
00-0082 DF-14		7.30E-1 3%			3.29E-3 4%	1.29E-4 5%	1.53E-5 12%
00-0083 PB * Process Blank	6.42E-3 13%	1.48E-1 6%	1.73E-3 6%	2.88E-3 5%	1.83E-3 7%	5.50E-4 10%	<1.E-5
00-0083 * Wash Solids	1.32E+1 2%	1.12E+3 3%	6.29E-1 4%	1.83E-1 6%	6.71E+0 4%	1.43E-1 6%	1.31E-2 15%
00-0083 DUP * Wash Solids	1.38E+1 2%	1.08E+3 3%	6.36E-1 4%	1.69E-1 6%	5.76E+0 4%	1.37E-1 7%	8.34E-3 24%
RPD	4%	4%	1%	8%	15%	4%	44%
Matrix Spike	94%	79%	105%		85%		
Blank Spike	108%	107%	110%		93%		
Blank	<2.E-5	<2.E-5	<3.E-6	<4.E-6	6.33E-6 30%	<8.E-7	<8.E-7

*Note: Results for the washed solids are reported per gram of dry weight.

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Radioanalytical Applications Team

00-0070

11/10/1999

Client : Hallen

Cognizant Scientist: LRGreenwood Date : 11/10/99

Concur : T Trang-le Date : 11/10/99

Procedure: PNL-ALO-450

Gamma Energy Analyses
Measured Activities (uCi/g) with 1-sigma error

ALO ID Client ID	Co-60 Error %	Cs-134 Error %	Cs-137 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %
PB-0070 Process Blank	<2.E-5	1.88E-5 43%	2.55E-4 7%	<6.E-5	<6.E-5	<6.E-5
00-0072 DF-20	5.15E-2 3%	<6.E-3	1.24E+2 2%	2.96E-2 11%	<8.E-2	<8.E-2
00-0072 DUP DF-20	5.22E-2 3%	<6.E-3	1.25E+2 2%	2.99E-2 12%	<9.E-2	<8.E-2
RPD	1%		1%	1%		
00-0073 DF-21	4.80E-2 3%	<6.E-3	1.22E+2 2%	2.73E-2 12%	<8.E-2	<8.E-2
00-0074 Wash Composite	4.97E-3 5%	<5.E-4	1.51E+1 2%	8.73E-4 46%	<2.E-2	<2.E-2
00-0074 DUP Wash Composite	5.26E-3 4%	<6.E-4	1.49E+1 2%	1.77E-3 22%	<2.E-2	<2.E-2
RPD	6%		2%	68%		
PB-0083 Process Blank	<3.E-3	3.58E-3 21%	7.48E-2 3%	<7.E-3	<7.E-3	<5.E-3
00-0083 * Wash Solids	4.43E-2 12%	<4.E-2	6.84E+1 2%	1.45E+1 2%	1.02E+1 2%	1.35E+1 4%
00-0083 DUP * Wash Solids	5.41E-2 10%	<4.E-2	7.02E+1 2%	1.38E+1 2%	9.66E+0 3%	1.34E+1 4%
RPD	20%		3%	5%	5%	1%

*Note: Results for the washed solids are reported per gram of dry weight.

RT HALLEN
Date 11/12/99
~~Route~~ T1-052
File
Copy file copy-record

PNNL Maintenance Services Record of Calibration

This record shall not be reproduced except in full without the written approval of the laboratory.

SYSTEM #: 03041

Uncal #	Model Number	MFR Name	Function	Serial #	Property #
03041	N/A	OMEGA	TEMP. DET.	325-427	N/A

RT HALLEN
Date 10/15/99
~~Route~~
File TI-052
Copy - File

Calibration Interval (Mo.)	Date of Calibration	Next Calibration Due	Building	Room	Area
24	09-15-99	09-2001	325 Bldg	601	300

CD TAG #	CAL #	Rev #
NA	21	6

Tolerance: ± 3°C of Reading

Calibration Standard # / Expiration Date
Traceable to National Institute of Standards and Technology (NIST)

0046 08-04-00
0097 09-15-99

Limitations: 0-100°C

Input/Standard Range: 0-100°C

Output/Indication Range: 0-100°C

Input	Standard Units	As Found	Desired Output	Units	As Found Error	Final Reading	Comments/Modifications, Repairs or Adjustments
0	°C	-.1	0	°C	-.1	-.1	
25	°C	22.9	25	°C	-2.1	22.9	New Calibration
50	°C	47.5	50	°C	-2.5	47.5	
75	°C	72.9	75	°C	-2.1	72.9	
100	°C	98.2	100	°C	-1.8	98.2	

COPY

Craftsman: Michael F. Teskey PNL 23 Calibration Date: 09-15-99 Acceptable: ✓

Acceptance of Data: Frances M Gordon Date: 10-5-99

Supervisor: [signature] Date: 9-24-99

Custodian: K. Brooks

Mail Stop: K6-24

Page 1 of 1

Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ...
ICPAES Data Report

Project: 29953
Client: R. T. Hallen

ACL Number(s): 00-0070 through 00-0083

Client ID: "DF-01" through "WASHED SOLIDS"

ASR Number: 5536

Total Samples: 14

Procedure: PNL-ALO-211, "Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectrometry" (ICP-AES).

Analyst: D.R. Sanders

Analysis Date (Filename):
11-01-99 (A0547, ALO-128),
11-02-99 (A0548, ALO-128),
11-03-99 (A0549, ALO-128),
11-04-99 (A0550, ALO-129),
11-12-99 (A0555, ALO-115 [KOH/Ni fusion])

See Chemical Measurement Center 98620: ICP-325-405-1 File for Calibration and Maintenance Records.

M&TE Number: ICPAES instrument -- WB73520
Mettler AT400 Balance -- Ser.No. 360-06-01-029

Jerry Wagner 12-7-99
Reviewed by

12-8-99
Concur

12/7/99

Thirteen radioactive liquid samples, DF-01 through DF-14 (ACL# 00-0070 through 00-0082), were analyzed by ICPAES after preparation by the Shielded Analytic Lab (SAL). Samples were prepared by SAL using PNL-ALO-128 acid digestion procedure. Approximately 5 to 6 grams of sample (weighed) was processed and diluted to a final volume of about 25ml. A 1ml aliquot taken from each processed sample was weighed and the density estimated by dividing the aliquot weight by the weight of water using the same pipette. The final volume of each processed sample was determined using the final weight of processed sample divided by the estimated density. Concentrations reported have been corrected for process and final volumes as reflected on "Corrected" bench sheets distributed by SAL.

One radioactive solid sample was prepared in duplicate by SAL, WASHED SOLIDS (ACL# 00-0083) using ALO-115 KOH/Ni fusion procedure. Prepared samples were analyzed by ICPAES. Approximately 0.2g aliquots were used to prepare samples using fusion procedure PNL-ALO-115 (KOH/Ni). Fusion prepared samples were diluted to a final volume of 100 ml. Additional dilution up to 10-fold was necessary during ICPAES analysis because of high manganese, strontium and sodium concentration. Fusion prepared samples required additional HCl. All solutions remained soluble after final dilution.

The one solid sample identified above, WASHED SOLIDS (ACL# 00-0083) was also prepared in duplicate using PNL-ALO-129 acid digestion procedure for solids by SAL and analyzed by ICPAES. Sample size was about 0.4g each. After digestion each sample was diluted to a final volume of approximately 20ml and weighed. A 1ml aliquot taken from each processed sample was weighed and the density estimated by dividing the aliquot weight by the weight of water using the same pipette. The final volume of each processed sample was determined using the final weight of processed sample divided by the estimated density. Relatively high analytical dilution was necessary because of very high concentration of manganese and strontium present in the sample and duplicate.

Measurement results reported have been corrected for preparation and analytical dilution. All results reported are in µg/g for the solids and liquid samples. Volumes and weights have been recorded on bench sheets and included with this report.

Specific analyte of interest requested is sodium for liquid samples (ALO# 00-0070 through 00-0082) prepared using PNL-ALO-128. Also requested were the following analytes in table-2 included with the ASR-5536 and special instructions. Analytes in table 2 include: Al, Ba, Ca, Cd, Co, Cr, Cu, Fe, K, La, Mg, Mn, Mo, Na, Ni, Pb, Si, Sr, Ti, U, and Zn.

The solid sample contained high concentrations (1 to 25 Wt%) of iron, manganese, sodium, and strontium. Concentration results for the fusion prepared and acid digestion prepared samples were similar and were typically within about 20% or better for analyte concentration above EQL.

The liquid samples contained high concentrations of sodium. A few samples had moderately high concentrations of aluminum. All other analytes measured were much lower in concentration.

Quality control check-standard results met tolerance requirements for analytes of interest except as noted below. Following is a list of quality control measurement results relative to ICPAES analysis tolerance requirements under MCS-033.

Five fold serial dilution:

(Solid samples/fusion) Results were within tolerance limit of ≤ 10% after correcting for dilution.

(Solid samples/acid dig.) Analytes of interest were within tolerance limit of ≤ 10% after correcting for dilution except as follows. Iron, calcium, chromium, and sodium were about 12 to 14% low after correcting for dilution. The reason may be related to high total dissolved solids (~ 0.4% TDS).

(Aqueous samples) All results for analytes of interest were within tolerance limit of ≤ 10% after correcting for dilution except sodium. Sodium concentration was slightly low (-12.9%) after dilution correction in sample 00-0082. The difference may be due to sodium carry-over from a previous sample containing high sodium.

Duplicate RPD (Relative Percent Difference):

(Solid samples/fusion) All analytes of interest were recovered within tolerance limit of ≤ 20% relative percent difference (RPD).

Duplicate RPD (Relative Percent Difference):

(Solid samples/acid dig.) All analytes of interest were recovered within tolerance limit of ≤ 20% relative percent difference (RPD) except sodium. Sodium was 20.9% RPD which is slightly above the tolerance limit of 20%. All other analyte concentrations were less than about 14% RPD, typically about 5% RPD or better.

(Aqueous samples)	All analytes of interest were recovered within tolerance limit of ≤ 20% relative percent difference (RPD).

Post-Spiked Samples (Group A):

(Solid samples/fusion)	All analytes of interest were recovered within tolerance of 75% to 125%.
(Solid samples/acid dig.)	All analytes of interest were recovered within tolerance of 75% to 125%.
(Aqueous samples)	All analytes of interest were recovered within tolerance of 75% to 125%.

Post-Spiked Samples (Group B):

(Solid samples/fusion)	All analytes of interest were recovered within tolerance of 75% to 125%.
(Solid samples/acid dig.)	All analytes of interest were recovered within tolerance of 75% to 125%.
(Aqueous samples)	All analytes of interest were recovered within tolerance of 75% to 125%.

Blank Spike:

(Solid samples/fusion)	A blank spike is not require for fusion prepared samples.
(Solid samples/acid dig.)	None prepared.
(Aqueous samples)	None prepared.

Matrix Spiked Sample:

(Solid samples/fusion)	A matrix spike is not require for fusion prepared samples.
(Solid samples/acid dig.)	None prepared.
(Aqueous samples)	None prepared.

Quality Control Check Standards (solid samples/fusion):

Concentration of all analytes of interest in the KOH/Ni fusion prepared analytical run was within tolerance limit of ± 10% accuracy in standards: QC_MCVA, QC_MCVB, and QC_SSTMCV except as follows. Potassium was high (~14%) in QC_MCVA two out of four times. Calibration Blank (ICP98.0) concentration was acceptable, less than two times IDL.

High Calibration Standard Check (solid samples/fusion):

Verification of the high-end calibration concentration for all analytes of interest is within tolerance of ± 5% accuracy except potassium. Potassium was slightly high (7% & 9%) in QC_SST however it was within 3% when measured in a single analyte standard of the same concentration.

Quality Control Check Standards (solid samples/acid dig.):

Concentration of all analytes of interest was within tolerance limit of ± 10% accuracy in standards: QC_MCVA, QC_MCVB. Several analytes were slightly above (11% to 15%) tolerance limits in check-standard QC_SSTMCV. The analytes of interest that were out of tolerance measured at the end of the run include iron, potassium, manganese, sodium, nickel, lead and silicon. Initially, all were within tolerance limits. Calibration Blank (ICP98.0) concentration was acceptable, less than two times IDL.

High Calibration Standard Check (solid samples/acid dig.):

Verification of the high-end calibration concentration for all analytes of interest is within tolerance of ± 5% accuracy except potassium, which measured 22% high at the end of the run. Potassium was not detected in the acid digested sample.

Quality Control Check Standards (aqueous samples):

Concentration of all analytes of interest in the acid digested aqueous prepared analytical run was within tolerance limit of ± 10% accuracy in the standards: QC_MCVA, QC_MCVB, and QC_SSTMCV except as follows. Potassium was slightly high, up to 13% in two of three QC_MCVA check standard measurements. The high potassium values may be due to memory effect or carry-over from one measurement to the next. Sodium was slightly high (11%) in QC_SSTMCV in one of two check standard measurements and may also be due to memory effect or carry-over from a sample with high sodium concentration.

High Calibration Standard Check (aqueous samples):

Verification of the high-end calibration concentration for all analytes of interest was within tolerance of ± 5% accuracy except potassium. Potassium concentration varied up to 11% too high in QC_SST check standard. Potassium concentration in aqueous samples usually was low when detected and then only slightly above EQL. Measurement of a single element potassium standard always came within tolerance limits.

Process Blank:

(Solid samples/fusion) All analytes of interest were within tolerance limit of ≤ EQL or < 5% of sample concentration in PNL-ALO-115 KOH/Ni fusion prepared samples.

(Solid samples/acid dig.) All analytes of interest were within tolerance limit of ≤ EQL or < 5% of sample concentration except silicon. Silicon concentration in the blank was equivalent to about 950 ug/g, which is equivalent to about 24% of the lowest concentration found in the samples. Silicon concentrations measured in the samples are generally below EQL.

(Aqueous samples) All analytes of interest were within tolerance limit of ≤ EQL or < 5% of sample concentration except iron and silicon. Silicon concentration in the process-blank was about the same concentration as the samples. Iron concentration found in the blank was variable. For one set of samples it was about the same concentration as the samples. In another processed batch of samples iron in the blank was much lower than in the samples. The reason for the variability between process blanks is not known.

<u>Laboratory Control Standard (LCS)</u>:

(Solid samples/fusion) All analytes of interest except strontium, at a concentration equal to or greater than EQL were recovered within tolerance limit of 75% to 125% in fusion prepared LCS standard. SRM-2710 Montana Soil was used for the LCS in PNL-ALO-115 fusion preparations. Strontium recovery was slightly above tolerance limit (126% recovery measured). Strontium concentration in the WASHED SOLIDS is about one hundred times higher in concentration than the LCS.

(Solid samples/acid dig.) No LCS was prepared for PNL-ALO-129 acid digested samples.

(Aqueous samples) No LCS was prepared for PNL-ALO-128 acid digested samples.

Analytes other than those requested by the client are for information only. Please note bracketed values listed in the data report are within ten times instrument detection limit and have a potential uncertainty much greater than 15%.

Comments:

1) "Final Results" have been corrected for all laboratory dilution performed on the sample during processing and analysis unless specifically noted.

2) Detection limits (DL) shown are for acidified water. Detection limits for other matrices may be determined if requested.

3) Routine precision and bias is typically ± 15% or better for samples in dilute, acidified water (e.g. 2% v/v HNO_3 or less) at analyte concentrations greater than ten times detection limit up to the upper calibration level. This also presumes that the total dissolved solids concentration in the sample is less than 5000 µg/mL (0.5 per cent by weight).

4) Absolute precision, bias and detection limits may be determined on each sample if required by the client.

5) The maximum number of significant figures for all ICP measurements is 2.

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

Det. Limit	Multiplier=	4.9	19.4	19.3	18.9	19.5
	ALO#=	00-0070-PB	00-0073 @5	00-0079 @5	00-0080 @5	00-0081 @5
	Client ID=	Process Blank	DF-21	DF-02	DF-03	DF-04
	Run Date=	11/2/99	11/2/99	11/2/99	11/2/99	11/2/99
(ug/mL)	(Analyte)	ug/g	ug/g	ug/g	ug/g	ug/g
0.025	Ag	--	--	--	--	--
0.060	Al	5.61	1,940	2,230	2,220	2,270
0.250	As	--	--	--	--	--
0.050	B	15.2	21.3	35.1	32.2	34.8
0.010	Ba	[0.17]	--	--	--	--
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	[1.7]	131	160	152	155
0.015	Cd	--	22.7	27.4	27.2	27.7
0.200	Ce	--	--	--	--	--
0.050	Co	--	[2.0]	[2.0]	[2.0]	[2.1]
0.020	Cr	--	36.5	7.23	6.65	6.89
0.025	Cu	--	16.3	12.1	12.0	12.3
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	[0.45]	8.60	6.49	6.96	7.40
2.000	K	--	615	730	716	732
0.050	La	--	--	--	--	--
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	--	[2.3]	15.1	20.0	17.7
0.050	Mo	--	12.5	14.8	14.8	15.0
0.150	Na	26.0	91,900	116,000	116,000	118,000
0.100	Nd	--	[2.7]	[3.1]	[2.7]	[2.9]
0.030	Ni	[0.40]	187	223	222	227
0.100	P	--	247	293	291	297
0.100	Pb	--	53.9	59.1	57.5	60.4
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	37.9	[32]	[43]	[38]	[40]
1.500	Sn	--	--	--	--	--
0.015	Sr	--	107	90.3	89.9	92.1
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	--	--	--
0.500	Tl	--	--	--	--	--
2.000	U	--	--	[41]	[39]	[42]
0.050	V	--	--	--	--	--
2.000	W	--	[62]	[73]	[73]	[74]
0.050	Y	--	[0.99]	[1.1]	[0.99]	[1.1]
0.050	Zn	[0.46]	[5.0]	[5.7]	[5.9]	[6.4]
0.050	Zr	--	[2.0]	[2.2]	[2.0]	[2.2]

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.
2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.
3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

Det. Limit	Multiplier=	19.2	18.8	19.7	19.7	19.4
	ALO#=	00-0070 @5	00-0071 @5	00-0072 @5	00-0072 DUP @5	00-0082 @5
	Client ID=	DF-01	DF-11	DF-20	DF-20	DF-14
	Run Date=	11/1/99	11/1/99	11/1/99	11/1/99	11/1/99
(ug/mL)	(Analyte)	ug/g	ug/g	ug/g	ug/g	ug/g
0.025	Ag	--	--	--	--	--
0.060	Al	2,140	2,220	1,990	2,050	1,910
0.250	As	--	--	--	--	--
0.050	B	39.2	32.5	19.8	19.1	25.5
0.010	Ba	--	--	--	--	--
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	169	157	140	144	140
0.015	Cd	26.3	27.1	23.4	24.0	22.8
0.200	Ce	--	--	--	--	--
0.050	Co	[2.0]	[2.1]	[2.1]	[2.1]	[1.9]
0.020	Cr	54.5	51.5	37.0	38.1	13.3
0.025	Cu	11.4	14.8	16.3	16.8	13.4
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	11.8	15.9	9.29	9.16	5.34
2.000	K	720	732	626	634	603
0.050	La	[1.4]	[1.1]	--	--	--
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	[2.4]	[3.3]	[2.4]
0.050	Mn	[1.9]	[1.8]	[2.3]	[2.4]	[3.9]
0.050	Mo	14.2	14.7	12.8	13.1	12.4
0.150	Na	112,000	119,000	102,000	110,000	105,000
0.100	Nd	[6.3]	[5.1]	[2.8]	[2.7]	[2.1]
0.030	Ni	215	222	194	200	189
0.100	P	282	294	253	259	246
0.100	Pb	57.9	65.9	54.9	56.0	55.1
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	102	[46]	[28]	[25]	[29]
1.500	Sn	--	--	--	--	--
0.015	Sr	161	153	113	118	94.4
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	--	--	--
0.500	Tl	--	--	--	--	--
2.000	U	[50]	[44]	[39]	--	[39]
0.050	V	--	--	--	--	--
2.000	W	[69]	[73]	[63]	[65]	[62]
0.050	Y	[2.0]	[1.6]	[1.0]	[1.0]	--
0.050	Zn	[5.7]	[6.0]	[5.7]	[6.0]	[5.5]
0.050	Zr	[3.8]	[3.4]	[2.1]	[2.1]	[1.5]

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.
2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.
3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

Det. Limit (ug/mL)	Multiplier= / ALO#= / Client ID= / Run Date= / (Analyte)	5.1 / 00-0074-PB / Process Blank / 11/3/99 / ug/g	4.8 / 00-0074 / Wash Composite / 11/3/99 / ug/g	4.7 / 00-0074 DUP / Wash Composite / 11/3/99 / ug/g	4.8 / 00-0075 / 1st. Wash / 11/3/99 / ug/g	23.1 / 00-0076 @5 / 2nd Wash / 11/3/99 / ug/g
0.025	Ag	--	[0.33]	[0.33]	[0.24]	--
0.060	Al	[0.52]	231	233	194	487
0.250	As	--	--	--	--	--
0.050	B	9.84	2.73	13.4	8.27	12.1
0.010	Ba	[0.051]	--	--	[0.048]	--
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	--	20.9	21.8	15.7	[42]
0.015	Cd	--	2.51	2.47	1.90	5.29
0.200	Ce	--	--	--	--	--
0.050	Co	--	--	--	--	--
0.020	Cr	[0.14]	2.24	2.02	1.19	[2.8]
0.025	Cu	--	1.76	1.75	1.38	[3.3]
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	1.51	1.55	[0.68]	[1.1]	[1.4]
2.000	K	--	[68]	[71]	[73]	[140]
0.050	La	--	--	--	--	--
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	--	[0.54]	[0.51]	[0.52]	--
0.050	Mo	--	[1.5]	[1.5]	[1.2]	[3.1]
0.150	Na	12.5	13,700	13,700	12,400	26,500
0.100	Nd	--	--	--	--	--
0.030	Ni	[0.50]	20.8	20.5	16.2	43.6
0.100	P	--	19.8	20.0	18.0	61.4
0.100	Pb	--	12.3	12.2	9.14	23.9
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	[12]	[15]	25.2	27.7	[18]
1.500	Sn	--	--	--	--	--
0.015	Sr	--	21.1	21.0	16.7	40.0
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	--	--	--
0.500	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.050	V	--	--	--	--	--
2.000	W	--	--	--	--	--
0.050	Y	--	--	--	--	--
0.050	Zn	--	[0.93]	[0.95]	[0.69]	[1.8]
0.050	Zr	--	--	--	--	--

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.
2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.
3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

Det. Limit (ug/mL)	Multiplier= / ALO#= / Client ID= / Run Date= / (Analyte)	4.9 / 00-0077 / 3rd Wash / 11/3/99 / ug/g	4.9 / 00-0078 / 4th Wash / 11/3/99 / ug/g			
0.025	Ag	[0.33]	[0.20]	--	--	--
0.060	Al	161	90.8	--	--	--
0.250	As	--	--	--	--	--
0.050	B	12.7	12.4	--	--	--
0.010	Ba	[0.080]	[0.063]	--	--	--
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	16.6	[10]	--	--	--
0.015	Cd	1.80	1.07	--	--	--
0.200	Ce	--	--	--	--	--
0.050	Co	--	--	--	--	--
0.020	Cr	2.52	1.77	--	--	--
0.025	Cu	[1.1]	[0.62]	--	--	--
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	3.44	[0.44]	--	--	--
2.000	K	[34]	[14]	--	--	--
0.050	La	--	--	--	--	--
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	[0.52]	[0.28]	--	--	--
0.050	Mo	[1.0]	[0.56]	--	--	--
0.150	Na	9,580	6,440	--	--	--
0.100	Nd	--	--	--	--	--
0.030	Ni	14.8	8.88	--	--	--
0.100	P	11.7	11.3	--	--	--
0.100	Pb	9.82	7.03	--	--	--
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	[18]	[10]	--	--	--
1.500	Sn	--	--	--	--	--
0.015	Sr	16.7	12.4	--	--	--
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	--	--	--
0.500	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.050	V	--	--	--	--	--
2.000	W	--	--	--	--	--
0.050	Y	--	--	--	--	--
0.050	Zn	[0.80]	[0.60]	--	--	--
0.050	Zr	--	--	--	--	--

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.
2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.
3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

Det. Limit (ug/mL)	Multiplier= / ALO#= / Client ID= / Run Date= / (Analyte)	50.5 / 00-0083-PB / Process Blank / 11/4/99 / ug/g	6177.8 / 00-0083 @125 / WASHED SOLIDS / 11/4/99 / ug/g	1256.0 / 00-0083-DUP @25 / WASHED SOLIDS / 11/4/99 / ug/g		
0.025	Ag	--	--	[97]	--	--
0.060	Al	[5.9]	8,500	8,400	--	--
0.250	As	--	--	--	--	--
0.050	B	--	--	--	--	--
0.010	Ba	[0.91]	[400]	387	--	--
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	--	[5,900]	5,750	--	--
0.015	Cd	--	--	[31]	--	--
0.200	Ce	--	[1,300]	[1,300]	--	--
0.050	Co	--	--	--	--	--
0.020	Cr	--	3,580	3,510	--	--
0.025	Cu	--	--	--	--	--
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	[4.4]	53,900	50,700	--	--
2.000	K	--	--	--	--	--
0.050	La	--	[810]	785	--	--
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	--	139,000	126,000	--	--
0.050	Mo	--	--	--	--	--
0.150	Na	--	71,300	91,200	--	--
0.100	Nd	--	[2,500]	2,380	--	--
0.030	Ni	[8.6]	--	[130]	--	--
0.100	P	--	[700]	[710]	--	--
0.100	Pb	--	[5,300]	5,360	--	--
0.750	Pd	--	--	[1,000]	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	948	[3,800]	[4,000]	--	--
1.500	Sn	--	--	--	--	--
0.015	Sr	--	289,000	280,000	--	--
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	[33]	--	--
0.500	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.050	V	--	--	--	--	--
2.000	W	--	--	--	--	--
0.050	Y	--	[320]	[320]	--	--
0.050	Zn	--	[330]	[310]	--	--
0.050	Zr	--	[2,300]	2,180	--	--

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.
2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.
3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

Det. Limit (ug/mL)					
	Multiplier=	1044.9	995.0	1100.1	
	ALO#=	00-0083-PB-Ni @2	00-0083-Ni @2	00-0083-DUP-Ni @2	
	Client ID=	*Process Blank*	*WASHED SOLIDS*	*WASHED SOLIDS*	
	Run Date=	11/12/99	11/12/99	11/12/99	
	(Analyte)	ug/g	ug/g	ug/g	
0.025	Ag	--	[100]	[120]	--
0.060	Al	[110]	7,770	7,260	--
0.250	As	--	--	--	--
0.050	B	--	[61]	[57]	--
0.010	Ba	[11]	321	314	--
0.010	Be	--	--	--	--
0.100	Bi	--	--	--	--
0.250	Ca	--	5,300	5,220	--
0.015	Cd	--	[34]	[36]	--
0.200	Ce	--	[560]	[490]	--
0.050	Co	--	--	--	--
0.020	Cr	--	3,100	3,170	--
0.025	Cu	--	[63]	[71]	--
0.050	Dy	--	--	--	--
0.100	Eu	--	--	--	--
0.025	Fe	[180]	44,300	43,400	--
0.050	La	--	644	633	--
0.030	Li	--	--	[34]	--
0.100	Mg	--	[190]	[200]	--
0.050	Mn	[100]	125,000	118,000	--
0.050	Mo	--	[67]	--	--
0.150	Na	[1,100]	71,900	72,700	--
0.100	Nd	--	1,820	1,670	--
0.100	P	--	[840]	[790]	--
0.100	Pb	--	3,790	4,600	--
0.750	Pd	--	--	--	--
0.300	Rh	--	--	--	--
1.100	Ru	--	--	--	--
0.500	Sb	--	--	--	--
0.250	Se	--	--	--	--
0.500	Si	--	5,740	[5,100]	--
1.500	Sn	--	--	--	--
0.015	Sr	--	255,000	242,000	--
1.500	Te	--	--	--	--
1.000	Th	--	--	--	--
0.025	Ti	--	[57]	[50]	--
0.500	Tl	--	--	--	--
2.000	U	--	[2,300]	[3,000]	--
0.050	V	--	--	--	--
2.000	W	--	--	--	--
0.050	Y	--	[260]	[260]	--
0.050	Zn	--	[310]	[300]	--
0.050	Zr	--	1,550	1,620	--

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.
2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.
3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

ALC-129 (Solids)

CORRECTED COPY
11/5/99

Pacific Northwest National Laboratory
Radiochemical Processing Laboratory
Shielded Facility Operation Team

SAMPLE PREP SHEET
(325 SHIELDED ANALYTICAL LABORATORY)

TI/ARF NO.: ASR5500.01 ~~ASR5536~~ PROJECT NO.: 29553 WBS NO.: ______

SAMPLE TYPE: WASHED SOLIDS

PREP TYPE: ACID DIGEST (ALO-129)

ISSUED BY: RT STEELE DATE: 10/15/99

CHAIN OF CUSTODY RQD: NONE

ANALYST: [signature] DATE: 10-28-99

QA PLAN: MCS-033 IMPACT LEVEL: ______

REVIEW: [signature] DATE: 10/29/99

TANK ID: AN107 / C104

CLIENT: RT HALLEN CORE ID: N/A

WORK PACKAGE NUMBER	ALO NUMBER	SAMPLE IDENTIFICATION:	ANALYTE OR ANALYSIS	SAMPLE WT	Sp.G. (g/mL)	WATER WT (g)	TOTAL VOL (mL)	SPIKE ID	SPIKE VOL (mL)	DILUTION FACTOR	DILUTION MATRIX	PIPET CALIB (mL)	MISC
W45525	00-0083-PB	PROCESS BLANK	ICP	0.4069			RTS ~~30~~	~~22.23~~	20.27				clear
	00-0083	WASHED SOLIDS		0.3963				~~34.37~~	20.11				yellow
	00-0083-DUP	WASHED SOLIDS		0.0731				~~24.25~~	19.91	RTS 11/5/99			yellow
W48489	99-2524-R	SUF-C104-010 (orange)		0.3704				~~23.30~~	20.31				yellow
	99-2524-DUP-R	SUF-C104-010 (Black)		0.3940				~~24.17~~	19.93				
	99-2525-R	SUF-C104-013 (orange)		0.3146				~~24.30~~	20.03				
	99-2525-Dup-R	SUF-C104-013 (Black)						~~24.39~~	20.00				

Warning! Contains .1 mL of "HF"

99-2524-R ... 99-2525-DUP-R Not all of the sample dissolved! A dark-black ppt. remained! [initials]

ACS5C 11-4-99 DRS

...INSTRUCTIONS & TABLE 2 FOR METALS LIST AND MRQs

Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory
Shielded Analytical Laboratory/Sample Receiving Laboratory

Corrected Copy

EXHIBIT I
PAGE 1 of 1

PNL-ALO-129 — HNO_3-HCl Acid Extraction of Solids for Metals Analysis Using a Dry-Block Heater

W48489

Client name: RT HALLEN
Work Auth. Doc (WAD): ASR 5536 / 5500.01
Tank/Core/Project: TANK AN107
Special instructions

Work package number: W45525
Project number: 29553
PNL QA plan: MCS-033
PNL impact level:
Prep. lab (SAL/SRPL/other): SAL
Preparation batch number:

	ACL Sample ID	ACL order number or Client sample ID	Vial Identifier	Vial weight (g)	Vial & sample weight (g)	Sample weight (g)	Total Vol. (ml)	~~Spike added Weight (g)~~	Gross Wt Digestate	1ml Digest Wt
1	00-0083-PB	PROCESS BLANK		8.2414	—	—	~~22.23~~	20.27	29.46845	1.0383
2	00-0083	WASHED SOLIDS		8.1015	8.50~~2~~784	0.4069	~~24.3706~~	20.11	30.2071	1.0932
3	00-0083-DUP	WASHED SOLIDS		8.1607	8.5570	0.3963	~~24.2538~~	19.91	30.1343	1.0945
4	99-2524-R	CUF-C104-010 (orange)		8.2268	8.2499	*0.0231	~~23.30~~	20.31	29.9799	1.0622
5	99-2524-DUP-R	CUF-C104-010 (Black)		8.2034	8.4738	0.2704	~~24.17~~	19.93	30.1518	1.0918
6	99-2525-R	CUF-C104-013 (orange)		8.0598	8.3838	*0.3240	~~24.30~~	20.03	30.1189	1.0923
7	99-2525-DUP-R	CUF-C104-013 (Black)		8.0893	8.4039	0.3146	~~24.39~~	20.00	30.1755	1.0950
8										
9		1 ml Pipet cal.	24°C				RTS 11/5/99			
10		.9847 $\bar{x}$ =	.9889							
11		.9836 s =	.0050							
12		.9898 RSD =	.5%							
13		.9903 Del.	.9916 ml							
14		.9960								

Analyst's sample preparation comments: After 1st heating, solids remained (dark). Added an additional ml of HNO_3 & HCl. Still had solids, so added 2 ml of HCl to each spl & heated again. Added .1 ml of HF to each! 00-0083 & Dup mostly went into solution, the other 4 did not.

* used All of spl.

Spike source:
PNL spike ID number:
Anal. balance M&TE: 360-06-01-016
Sample filtered (yes/no): yes.

(1) Process factor = Final volume (ml) / [Vial & sample weight (g) - Vial weight (g)]

Other sample preparation worksheets may be substituted at the discretion of the Cognizant Scientist. Use one worksheet per client.

Analyst/Date: [signature] 10-28-99 Reviewer/Date: [signature] 10/29/99

Pacific Northwest National Laboratory
Radiochemical Processing Laboratory
Shielded Facility Operation Team

SAMPLE PREP SHEET
(325 SHIELDED ANALYTICAL LABORATORY)

TI/ARF NO.: ASR5536 PROJECT NO.: 29553 WBS NO.: ______ SAMPLE TYPE: WASHED SOLIDS

ISSUED BY: RT STEELE DATE: 10/15/99 PREP TYPE: Ni/KOH FUSION (ALO-115)

ANALYST: [signature] DATE: 10-26-99 CHAIN OF CUSTODY RQD: NONE

REVIEW: Richard L. Steele DATE: 11/1/99 QA PLAN: MCS-033 IMPACT LEVEL: ______

CLIENT: RT HALLEN CORE ID: N/A TANK ID: AN107

WORK PACKAGE NUMBER	ALO NUMBER	SAMPLE IDENTIFICATION	ANALYTE OR ANALYSIS	SAMPLE WT	Sp.G.(A) (g/mL)	WATER WT (g)	TOTAL VOL (mL)	SPIKE ID	SPIKE VOL (mL)	DILUTION FACTOR	DILUTION MATRIX	PIPET CALIB (mL)	MISC
W45525	00-0083-PB	PROCESS BLANK	1,2				100						
	00-0083	WASHED SOLIDS	1,2	.2010									
	00-0083-DUP	WASHED SOLIDS	1,2	.1818									
	SRM 2710	LCS/00-0083/Ni	1	.2335									

(1) REQUIRED ANALYSES:
ICP

(2) REQUIRED ANALYSES:
GEA, SR-90, ICP/MS (Tc-99), Am/Cm, Pu/AEA, ALPHA/GROSS

(A) Specific Gravity of ORIGINAL material. **SEE ASR5536 SPECIAL INSTRUCTIONS & TABLE 2 FOR ANALYTE LIST AND MRQs**

Lori,
ICP samples were delivered ...

Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory
Shielded Analytical Laboratory

PNL-ALO-115

Solubilization of Metals from Solids Using a KOH-KNO3 Fusion

Client name: RT HALLEN
Work Auth. Doc (WAD): ASR 5536
Tank/Core/Project: TANK AN107
Special instructions

Work package number: W45525
Project number: 29553
PNL QA plan: MCS-033
PNL impact level:
Prep. lab (SAL/SRPL/other): SAL
Preparation batch number:

	ACL Sample ID	ACL order number or Client sample ID	Crucible Identifier	Crucible weight (g)	Crucible + sample weight (g)	Sample weight (g)	Spike added Vol. (ml)	Spike added Weight. (g)	Final solution Volume (ml)	Process Factor (1)
1	00-0083-PB	PROCESS BLANK							100	
2	00-0083	WASHED SOLIDS	1	33.1673	33.3083	0.2010				497.51
3	00-0083-DUP	WASHED SOLIDS	2	33.6686	33.8504	0.1818				550.06
4	SRM 2710	LCS/00-0083/Ni	3	33.8283	34.0618	0.2335				428.27
5										
6										
7										
8										
9										
10										
11										
12										
13										
14										

Analyst's sample preparation comments:

DOSE RATE:

Spike source:
PNL spike ID number:
Anal. balance M&TE: 360.06.01.016

HCl volume added (ml): 2 ml
Solution heated (yes/no): yes
Sample filtered (yes/no): no

(1) Process factor = Final volume (ml) / [Crucible & sample weight (g) - Crucible weight (g)]

Other sample preparation worksheets may be substituted at the discretion of the Cognizant Scientist. Use one worksheet per client.

Rev. 2.0 7-28-95 JMR

Analyst/Date: [signature] 10-26-99 Reviewer/Date: [signature] 11/1/99

Pacific Northwest National Laboratory
Radiochemical Processing Laboratory
Shielded Facility Operation Team

CORRECTED COPY
11/5/99

SAMPLE PREP SHEET
(325 SHIELDED ANALYTICAL LABORATORY)

TI/ARF NO.: ASR5536 PROJECT NO.: 29553 WBS NO.: ______ SAMPLE TYPE: AQUEOUS

ISSUED BY: RT STEELE DATE: 10/14/99 PREP TYPE: ACID DIGEST (ALO-128)

ANALYST: [signature] DATE: 10-19-99 CHAIN OF CUSTODY RQD: NONE

REVIEW: [signature] DATE: 10-19-99 QA PLAN: MCS-033 IMPACT LEVEL: ______

CLIENT: RT HALLEN CORE ID: N/A TANK ID: AN107

WORK PACKAGE NUMBER	ALO NUMBER	SAMPLE IDENTIFICATION	ANALYTE OR ANALYSIS	SAMPLE WT	Sp.G.(A) (g/mL)	WATER WT (g)	TOTAL VOL (mL)	SPIKE ID	SPIKE VOL (mL)	DILUTION FACTOR	DILUTION MATRIX	PIPET CALIB (mL)	MISC
W45525	00-0070-PB	PROCESS BLANK	2	4.8715			~~25.18~~	23.75					
	00-0070	DF-01	1	6.3288			~~25.97~~	24.36					
	00-0071	DF-11	1	6.4425			~~25.93~~	24.16					
	00-0072	DF-20	2	6.2462			~~25.71~~	24.58					
	00-0073	DF-21	2	6.3423			~~27.75~~	24.62	RCS				
	00-0079	DF-02	1	6.4473			~~24.68~~	24.91	11/5/99				
	00-0080	DF-03	1	6.5701			~~27.75~~	24.78					
	00-0081	DF-04	1	6.3495			~~27.44~~	24.81					
	00-0082	DF-14	1	6.3427			~~25.58~~	24.22					
	00-0072-DUP	DF-20	2	6.2764			~~25.67~~	24.75					
	00-0074 00-0074.Dup	WASH COMPOSITE	2 (ICP DONE)	5.1441 5.1951			~~26.35~~ ~~26.35~~	24.65 24.53					

(1) REQUIRED ANALYSES:
Sr-90, Am/Cm, ICP Na (INCLUDE ALL ICP)

(2) REQUIRED ANALYSES:
GEA, SR-90, ICP/MS (Tc-99), Am/Cm, Pu/AEA, ALPHA/GROSS, ICP

(A) Specific Gravity of ORIGINAL material. SEE ASR5536 SPECIAL INSTRUCTIONS & TABLE 2 FOR ANALYTE LIST AND MRQs

Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory

Corrected Copy

EXHIBIT I
PAGE 1 of 1

PNL-ALO-128

Nitric and Hydrochloric Acid Extraction of Liquids Using a Dry-Block Heater

Client name: RT Hallen
Work Auth. Doc (WAD): ASR 5536
Tank/Core/Project: Tank AN107
Special instructions

Work package number: W45525
Project number: 29553
PNL QA plan: MCS-033
PNL impact level:
Prep. lab (SAL/SRPL/other): SAL
Preparation batch number:

	ACL Sample ID	Client sample ID	Gross Weight (g)	Tare Weight (g)	Net Sample Weight (g)	Sample Volume (ml)	Final Soln Gross Wgt (g)	Final Soln 1mL Wgt (g)	Final Volume (ml)
1	00-0070-PB	PROCESS BLANK (DIW)	33.6851	28.7936	4.8915	4.9931	53.2503	1.0208	~~25.18~~ 23.7
2	00-0070	DF-01	34.9260	28.5972	6.3288		55.1665	1.0811	~~25.97~~ 24.3
3	00-0071	DF-11	35.0609	28.6184	6.4425		55.0539	1.0850	~~28.93~~ 24.1
4	00-0072	DF-20	34.8548	28.6086	6.2462		55.1747	1.0715	~~28.71~~ 24.
5	00-0073	DF-21	35.0557	28.7134	6.3423		55.5024	1.0786	~~29.15~~ 24
6	00-0079	DF-02	35.1893	28.~~7420~~ 7420	6.4473		55.9337	1.0822	~~29.68~~ 24.
7	00-0080	DF-03	35.3353	28.7652	6.5701		55.7821	1.0807	~~29.45~~ 24.
8	00-0081	DF-04	34.8761	28.5266	6.3495		55.5532	1.0799	~~29.44~~ 24
9	00-0082	DF-14	34.8430	28.6003	6.2427		54.9610	1.0770	~~28.58~~ 24.
10	00-0072 -DUP	DF-20	34.8967	28.6203	6.2764		55.2600	1.0671	~~28.67~~ 24.
11									RTS 11/5/99
12									
13									
14									

Analyst's sample preparation comments:

5mL pipet	DIW @ 26°C	1mL pipet	DIW @ 26°C
4.9669	4.9771	.9884	.9882
4.9682	.0116	.9889	.0005
4.9736	.23%	.9882	.05%
4.9950	Cal. = 4.9931 ml	.9875	Cal. = .9914 ml
4.9516		.9880	

Spike source:
PNL spike ID number:
Anal. balance M&TE: 360-06-01-016
Sample filtered (yes/no): no

Other sample preparation worksheets may be substituted at the discretion of the Cognizant Scientist. Use one worksheet per client.

Analyst/Date: [signature] 10-15-99 Reviewer/Date: [signature] 10/19/99

Pacific Northwest National Laboratory
Radiochemical Processing Laboratory
Shielded Facility Operation Team

Corrected Copy
11/5/99

SAMPLE PREP SHEET
(325 SHIELDED ANALYTICAL LABORATORY)

TI/ARF NO.: ASR5536 PROJECT NO.: 29553 WBS NO.: ______ SAMPLE TYPE: AQUEOUS WASH SOLUTIONS

ISSUED BY: RT STEELE DATE: 10/12/99 PREP TYPE: ACID DIGEST (ALO-128)

ANALYST: [illegible] DATE: 10-14-99 CHAIN OF CUSTODY RQD: NONE

REVIEW: [illegible] DATE: 10-14-99 QA PLAN: MCS-033 IMPACT LEVEL: ______

CLIENT: RT HALLEN CORE ID: N/A TANK ID: AN107

WORK PACKAGE NUMBER	ALO NUMBER	SAMPLE IDENTIFICATION	ANALYTE OR ANALYSIS	SAMPLE WT	Sp.G.(1) (g/mL)	WATER WT (g)	TOTAL VOL (mL)	SPIKE ID	SPIKE VOL (mL)	DILUTION FACTOR	DILUTION MATRIX	PIPET CALIB (mL)	MISC
W45525	00-0074-PB	PROCESS BLANK	ICP	4.9964			~~27.24~~	25.30					
	00-0074	WASH COMPOSITE		5.1441			~~26.35~~	24.65					
	00-0074-DUP	WASH COMPOSITE		5.1951			~~26.35~~	24.53	RGS				
	00-0075	1ST WASH		5.1756			~~26.79~~	24.61	11/5/99				
	00-0076	2ND WASH		5.3331			~~26.76~~	24.58					
	00-0077	3RD WASH		5.1150			~~26.96~~	25.23					
	00-0078	4TH WASH		5.0790			~~26.64~~	24.94					

(1) Specific Gravity of ORIGINAL material. SEE ASR5536 SPECIAL INSTRUCTIONS & TABLE 2 FOR METALS LIST AND MRQs

Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory

PNL-ALO-128

Nitric and Hydrochloric Acid Extraction of Liquids Using a Dry-Block Heater

Client name: RT Hallen
Work Auth. Doc (WAD): ASR 5536
Tank/Core/Project: Tank AN107
Special instructions

Work package number: W45525
Project number: 29553
PNL QA plan: MCS-033
PNL impact level:
Prep. lab (SAL/SRPL/other): SAL
Preparation batch number:

	ACL Sample ID	Client sample ID	Gross Weight (g)	Tare Weight (g)	Net Sample Weight (g)	Sample Volume (ml)	Final Soln Gross Wgt (g)	Final Soln 1mL Wgt (g)	Final Volume (ml)
1	00-0074-PB	PROCESS BLANK	30.4720	25.4756	4.9964	5.0004 *	51.7529	1.0285	~~27.39042~~ 25.3
2	00-0074	WASH COMPOSITE	33.67~~24~~93	28.5352	5.~~[illegible]~~1441		54.0238	1.0238	~~26.3508~~ 24.6
3	00-0074-DUP	WASH COMPOSITE	34.0347	28.8396	5.1951		54.2619	1.0264	~~26.3490~~ 24.5
4	00-0075	1ST WASH	33.2464	28.0714	5.1750		53.7493	1.0331	~~26.7877~~ 24.0
5	00-0076	2ND WASH	29.9396	24.6165	5.3231		50.2644	1.0334	~~26.7642~~ 24.
6	00-0077	3RD WASH	29.8782	24.7632	5.1150		50.8460	1.0237	~~26.9625~~ 25.
7	00-0078	4TH WASH	29.3939	24.3149	5.0790		50.0897	1.0236	~~26.6415~~ 24.9
8									RTS 11/5/99
9									
10									
11									
12									
13									
14									

Analyst's sample preparation comments: 1 mL Pipet 26°C

* Pipet Calib. on Composite Bench Sheet .9858 $\bar{x}$ = .9871
.9862 s = .0014
.9878 RSD = .14%
.9892 $\boxed{\text{Del} = .9903 \text{ mL}}$
.9864

Spike source:
PNL spike ID number:
Anal. balance M&TE: 360-06-01-016
Sample filtered (yes/no): NO

Other sample preparation worksheets may be substituted at the discretion of the Cognizant Scientist. Use one worksheet per client.

Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ...
ICPAES Data Report

Project: 29953
Client: R.T. Hallen & K.P. Brooks

ACL Number(s): 99-2524-R through 99-2525-DUP-R
[re-runs]

Client ID: "CUF-C104-010(Orange)" through "CUF-C104-013(Black)"

ASR Number: 5500.01

Total Samples: 4

Procedure: PNL-ALO-211, "Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectrometry" (ICP-AES).

Analyst: D.R. Sanders

Analysis Date (Filename): 11-04-99 (A0550)

See Chemical Measurement Center 98620: ICP-325-405-1 File for Calibration and Maintenance Records.

M&TE Number: ICPAES instrument -- WB73520
Mettler AT400 Balance -- Ser.No. 360-06-01-029

Jerry Wagner 1-18-00
Reviewed by

M W Urie 1-19-00
Concur

RT HALLEN
Date 1/21/00
~~Route~~
File TJ-052
Copy Record

Four radioactive solid samples **CUF-C104-010(Orange)** through **CUF-C104-013(Black)** (ACL# 99-2524-R through 99-2525-DUP-R) were prepared by SAL using ALO-129 acid digestion of solids procedure. Prepared samples were analyzed by ICPAES. Approximately 0.02g to 0.32g aliquots were processed and diluted to a final volume of about 20 ml (weighed). Some dark-black residue remained insoluble from all the samples after processing. The final volume of each processed sample was determined using the final weight of processed sample divided by an estimated density. Analytical dilution of 5-fold to 50-fold was required because of high concentration of aluminum, iron, manganese, sodium, thorium, uranium, and zirconium. Sample **CUF-C104-010(Orange)** was prepared with only about one-tenth the amount of solids compared with the other three samples. As a result, only a 5-fold analytical dilution was required.

The concentration of palladium reported previously appears to be an artifact caused by spectral interference particularly from high concentrations of thorium and uranium. Because of this, the actual concentration of palladium is too low to determine by ICPAES without chemical separation of the interfering analytes. Sodium in sample **CUF-C104-010(Orange)** appeared to be about ten times higher than the other three samples. The reason for the discrepancy is not known.

Measurement results reported have been corrected for preparation and analytical dilution. All results reported are in µg/g for the solids samples. Volumes and weights have been recorded on bench sheets and included with this report.

Quality control check-standard results met tolerance requirements for analytes of interest except as noted below. Following is a list of quality control measurement results relative to ICPAES analysis tolerance requirements under MCS-033.

Five fold serial dilution:

(Solid samples/acid dig.) Analytes of interest were within tolerance limit of ≤ 10% after correcting for dilution except silver, magnesium and uranium. Because of the very high concentration of thorium, interference correction to silver, magnesium and uranium were incorrect leading to inaccurate concentration values in the 5-fold diluted sample. At 25-fold and 50-fold dilution all analytes of interest were within ≤ 10% after correcting for dilution.

Duplicate RPD (Relative Percent Difference):

(Solid samples/acid dig.) All analytes of interest were recovered within tolerance limit of ≤ 20% relative percent difference (RPD) except silicon in ACL# 99-2524-R and its duplicate analyzed at 5-fold dilution. As noted earlier the reason for the large (150% RPD) for silicon is not known.

Post-Spiked Samples (Group A):

(Solid samples/acid dig.) All analytes of interest were recovered within tolerance of 75% to 125%.

Post-Spiked Samples (Group B):

(Solid samples/acid dig.) All analytes of interest were recovered within tolerance of 75% to 125%.

Blank Spike:

(Solid samples/acid dig.) None prepared.

Matrix Spiked Sample:

(Solid samples/acid dig.) None prepared.

Quality Control Check Standards (solid samples/acid dig.):

Concentration of all analytes of interest was within tolerance limit of ± 10% accuracy in standards: QC_MCVA.

Tin and thorium were low (-15% and –61% respectively) in QC_MCVB. Single element standards of 2 ppm tin and 10 ppm thorium were measured separately and were within the tolerance limits.

Several analytes: iron, potassium, manganese, sodium, nickel, lead, silicon and zirconium were a little high (11% to 15%) and out of tolerance limits in check-standard QC_SSTMCV.

Calibration Blank (ICP98.0) concentration was acceptable, less than two times IDL.

High Calibration Standard Check (solid samples/acid dig.):

Verification of the high-end calibration concentration for all analytes of interest is within tolerance of ± 5% accuracy except potassium, which measured 22% high at the end of the run. Potassium was not detected in any of the samples.

Process Blank:

(Solid samples/acid dig.) All analytes of interest were within tolerance limit of ≤ EQL or < 5% of sample concentration except silicon. Silicon concentration in the blank was equivalent to about 1300 ug/g. Silicon at about 25,000 µg/g was only found in sample **CUF-C104-010 (Orange)** (ACL# 99-2524-R).

Laboratory Control Standard (LCS):

(Solid samples/acid dig.) No LCS was prepared for PNL-ALO-129 acid digested samples.

Analytes other than those requested by the client are for information only. Please note bracketed values listed in the data report are within ten times instrument detection limit and have a potential uncertainty much greater than 15%.

Comments:

1) "Final Results" have been corrected for all laboratory dilution performed on the sample during processing and analysis unless specifically noted.

2) Detection limits (DL) shown are for acidified water. Detection limits for other matrices may be determined if requested.

3) Routine precision and bias is typically ± 15% or better for samples in dilute, acidified water (e.g. 2% v/v HNO_3 or less) at analyte concentrations greater than ten times detection limit up to the upper calibration level. This also presumes that the total dissolved solids concentration in the sample is less than 5000 µg/mL (0.5 per cent by weight).

4) Absolute precision, bias and detection limits may be determined on each sample if required by the client.

5) The maximum number of significant figures for all ICP measurements is 2.

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

Det. Limit (ug/mL)	Multiplier= / ALO#= / Client ID= / Run Date= / (Analyte)	4396.1 / 99-2524-R @5 / CUF-C104-010 (Orange) / 11/4/99 / ug/g	1842.6 / 99-2524-DUP-R @25 / CUF-C104-010 (Black) / 11/4/99 / ug/g	1545.5 / 99-2525-R @25 / CUF-C104-013 (Orange) / 11/4/99 / ug/g	1589.3 / 99-2525-DUP-R @25 / CUF-C104-013 (Black) / 11/4/99 / ug/g
0.025	Ag	[900]	1,060	1,850	1,650
0.060	Al	144,000	165,000	40,100	39,400
0.250	As	--	--	--	--
0.050	B	[1,300]	--	[77]	[80]
0.010	Ba	[210]	226	412	472
0.010	Be	--	[49]	[69]	[66]
0.100	Bi	--	--	--	--
0.250	Ca	[4,900]	5,210	8,830	9,410
0.015	Cd	854	978	1,730	1,830
0.200	Ce	--	--	[420]	--
0.050	Co	--	--	--	--
0.020	Cr	1,620	1,830	2,130	2,190
0.025	Cu	[210]	[270]	485	523
0.050	Dy	--	--	--	--
0.100	Eu	--	--	--	--
0.025	Fe	46,000	52,500	91,300	97,100
2.000	K	--	--	--	--
0.050	La	--	[140]	[250]	[290]
0.030	Li	[470]	587	513	536
0.100	Mg	[910]	[990]	1,570	1,710
0.050	Mn	10,200	11,600	20,300	21,600
0.050	Mo	--	--	--	--
0.150	Na	33,400	36,100	68,700	63,800
0.100	Nd	--	[290]	[560]	[600]
0.030	Ni	2,990	3,350	5,860	6,230
0.100	P	5,080	2,510	[1,000]	[1,200]
0.100	Pb	[1,600]	1,870	3,170	3,350
0.750	Pd	--	--	[3,700]	--
0.300	Rh	--	--	--	--
1.100	Ru	--	--	--	--
0.500	Sb	--	--	--	--
0.250	Se	--	--	--	--
0.500	Si	25,200	[2,500]	[1,700]	[1,700]
1.500	Sn	--	--	--	--
0.015	Sr	[110]	[110]	[190]	[210]
1.500	Te	--	--	--	--
1.000	Th	[5,900]	73,600	126,000	134,000
0.025	Ti	[160]	[170]	[300]	[320]
0.500	Tl	--	--	--	--
2.000	U	[42,000]	52,600	93,500	100,000
0.050	V	--	--	[80]	--
2.000	W	--	--	--	--
0.050	Y	--	--	--	--
0.050	Zn	[340]	[300]	[650]	[760]
0.050	Zr	60,600	64,600	113,000	123,000

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.
2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.
3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

Det. Limit (ug/mL)	Multiplier= / ALO#= / Client ID= / Run Date= / (Analyte)	50.5 / 00-0083-PB / Process Blank / 11/4/99 / ug/g	6177.8 / 00-0083 @125 / WASHED SOLIDS / 11/4/99 / ug/g	1256.0 / 00-0083-DUP @25 / WASHED SOLIDS / 11/4/99 / ug/g		
0.025	Ag	--	--	[97]	--	--
0.060	Al	[5.9]	8,500	8,400	--	--
0.250	As	--	--	--	--	--
0.050	B	--	--	--	--	--
0.010	Ba	[0.91]	[400]	387	--	--
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	--	[5,900]	5,750	--	--
0.015	Cd	--	--	[31]	--	--
0.200	Ce	--	[1,300]	[1,300]	--	--
0.050	Co	--	--	--	--	--
0.020	Cr	--	3,580	3,510	--	--
0.025	Cu	--	--	--	--	--
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	[4.4]	53,900	50,700	--	--
2.000	K	--	--	--	--	--
0.050	La	--	[810]	785	--	--
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	--	139,000	126,000	--	--
0.050	Mo	--	--	--	--	--
0.150	Na	--	71,300	91,200	--	--
0.100	Nd	--	[2,500]	2,380	--	--
0.030	Ni	[8.6]	--	[130]	--	--
0.100	P	--	[700]	[710]	--	--
0.100	Pb	--	[5,300]	5,360	--	--
0.750	Pd	--	--	[1,000]	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	948	[3,800]	[4,000]	--	--
1.500	Sn	--	--	--	--	--
0.015	Sr	--	289,000	280,000	--	--
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	[33]	--	--
0.500	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.050	V	--	--	--	--	--
2.000	W	--	--	--	--	--
0.050	Y	--	[320]	[320]	--	--
0.050	Zn	--	[330]	[310]	--	--
0.050	Zr	--	[2,300]	2,180	--	--

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.
2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.
3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

Battelle PNNL/RPG/Inorganic Analysis --- IC Report

REVISION 01 – Addition of 00-0083 Process Blank Results

Client: R. Hallen **Charge Code/Project:** W45525 / 29953
ACL Numbers: 00-0072 to -0074, -0083 **ASR Number:** 5536
Analyst: MJ Steele **Analysis Date:** November 01-03,11, 1999

Procedure: PNL-ALO-212, "Determination of Inorganic Anions by Ion Chromatography"
M&TE: IC system (WD25214); Balance (360-06-01-031) --- See Chemical Measurement Center 98620 RIDS IC File for Calibration, Standards Preparations, and Maintenance Records.

Final Results:

Lab ID	Sample ID	Hot Cell Dil Fctr	F µg/g	Cl µg/g	NO_2 µg/g	Br µg/g	NO_3 µg/g	PO_4 µg/g	SO_4 µg/g	C_2O_4 µg/g
00-0072	DF-20	8.02	< 2000	< 2000	21,800	< 2000	86,700	< 4000	< 4000	< 4000
	00-0072 MS Rec		113%	101%	109%	109%	115%	111%	114%	111%
00-0073	DF-21	8.02	< 2000	< 2000	21,600	< 2000	88,100	< 4000	< 4000	< 4000
00-0074	Wash Comp	4.95	< 125	< 125	2,900	< 125	11,600	< 250	480	3,600
00-0074 Dup	Wash Comp Dup	4.91	< 125	< 125	2,700	< 125	10,700	< 250	460	3,300
	RPD		n/a	n/a	8%	n/a	7%	n/a	6%	8%
00-0083PB	Process Blank	102.0*	< 25	< 25	< 50	< 25	< 50	< 50	< 50	< 50
00-0083	Washed Solids	114.0	< 300	< 300	1,800	< 300	8,000	< 600	1,200	25,500
00-0083 Dup	Washed Solids Dup	89.91	< 250	400	1,700	< 250	8,700	< 500	1,200	21,100
	RPD		n/a	n/a	8%	n/a	9%	n/a	1%	19%
	00-0083 MS Rec		102%	99%	102%	105%	98%	104%	105%	99%

RPD = Relative Percent Difference (between sample and duplicate/replicate)
MS Rec = Matrix Spike Standard % recovery
* = average 00-0083 hot cell dilution factor to provide process blank results in µg/g.

The samples were analyzed by ion chromatography (IC) for inorganic anions as specified in the governing ASR. The liquid samples were diluted at the IC workstation from 100-fold to 1,000-fold to ensure that all anions were within the calibration range. The liquid samples are reported in µg/g of liquid and the solids samples are reported in µg/g of dried solids.

Q.C. Comments:

Duplicates: Duplicate preparations from the hot cells were provided to the IC laboratory for both the liquid and solids matrices; i.e., samples "Wash Comp" and "Washed Solids", respectively. The relative percent differences (RPD) between replicates are within the acceptance criteria of 20% for all anions measured above the EQL.

Matrix Spike: Matrix spikes were prepared and measured for samples "DF-20" and "Washed Solids". The spike recoveries for all anions are within the 75% to 125% recovery acceptance criteria.

RT HALLEN
Date 11/19/99
Route
File T1-052
Copy Record

Blank Spike: Although no blank spike, which is used as the laboratory control sample, was analyzed directly with these samples, three blank spike samples were analyzed with each of the daily runs. All anions for the blank spikes recovered within the acceptance criteria of 80% to 120%.

System Blank/Processing Blanks: Approximately ten system blanks were process during the analysis of the samples. With the exception of only single nitrate value, no anions were detected above reportable concentrations in the system blanks. Since the nitrate results are high, this single QC failure does not affect the reported nitrate results.

Quality Control Calibration Verification Check Standards: Approximately ten mid-range verification standards were analyzed throughout the analysis runs. Except for a single phosphate value, the reported results for all analytes of interest were recovered within the acceptance criteria of ±10% for the verification standard. The one phosphate result recovered at +11% above the true value. No phosphate was detected in any of the samples, thus the single phosphate failure has no impact on the reported results.

General Comments:

- The reported "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
- The low calibration standards are defined as the estimated quantitation limit (EQL) for the reported results and assume non-complex aqueous matrices. Actual detection limits or quantitation limits for specific sample matrices may be determined, if requested.
- Routine precision and bias are typically ±15% or better for non-complex aqueous samples that are free of interference and have similar concentrations as the measured anions.

Analyst: MJ Steele **Date** 11/16/99

Approval: MW Urie **Date** 11/16/99

Archive Information:

Files: ASR 5533 Lumetta.doc	ASR 5463 5533 -36 -68 -71.xls

Battelle
Pacific Northwest Laboratories

Project Number

Internal Distribution

329/4 File
Mike Urie

Date November 5, 1999

To Richard Hallen

From Tom Farmer

Subject ICP/MS Analysis of Submitted Samples
(ALO#00-0070,0072-0074 and 00-0083)

The previously reported sample results were incorrect due to an error in the dilution volumes received from Rick Steele. Here are the corrected results using the correct dilution volumes.

James

RT HALLEN
Date 11/10/99
~~Route~~
File T1-052
Copy File Copy

11/5/99

Hallen Tc-99 Analysis

November 4, 1999(Revised 11/5/99)

Results are reported in µg analyte/g (ppm) of original sample.
The uncertainty of the results is estimated at ±10%.

Sample ID		ICP/MS Number	Tc-99 µg/g	*Ru-101 µg/g
1%HNO3		9b03a1	<0.0002	
1%HNO3		9b03a6	<0.0002	
1%HNO3		9b03a18	<0.0002	
00-0070-PB	Process Blank	9b03a8	<0.01	0.001
00-0072	DF-20	9b03a14	2.59	4
00-0072-DUP	DF-20	9b03a15	2.56	4
00-0073	DF-21	9b03a16	2.86	5
00-0073 + spike	DF-21	9b03a17	3.33	
Spike Recovery			**103%**	
00-0074	Wash Composite	9b03a12	0.293	0.5
00-0074-DUP	Wash Composite	9b03a13	0.287	0.5
00-0083-PB	Process Blank	9b03a7	<0.5	0.01
00-0083	Washed Solids	9b03a10	6.73	5
00-0083-DUP	Washed Solids	9b03a11	10.5	7
SRM 2710	LCS/00-0083/Ni	9b03a9	<0.5	0.03
CCV results are reported in ng/ml (ppb)				
2ppb Tc-99 CCV		9b03a3	2.19	
2ppb Tc-99 CCV		9b03a19	2.14	
100ppb Co		9b03a20	<0.0002	

DATA REVIEW

Reviewed by:

Date: 5Nov99 Pages: 1 of 1

Battelle PNNL/RPG/Inorganic Analysis --- TOC/TIC Report

Client: R. Hallen **Charge Code/Project:** W45525 / 29953

ACL Numbers: 00-0072 to 00-0074, 00-0083 **ASR Number:** 5536
Analyst: MJ Steele **Analysis Date:** December 9-10, 1999

Procedure: PNL-ALO-381, "Direct Determination of TC, TOC, and TIC in Radioactive Sludges and Liquids by Hot Persulfate Method"

RT HALLEN
Date 1/18/2000
Route -
File - TT-52
Copy - record

M&TE: Carbon System (WA92040); Balance (360-06-01-023).

Final Results:

Lab Number	Sample ID	Density ug/ml[1]	Hot Cell Dil Fctr	TIC		TOC		TC	
				ug/g	RPD(%)	ug/g	RPD(%)	ug/g	RPD(%)
Liquids									
00-0072 PB	Process Blank		6.47	<40		<120		--	
00-0072	DF-20	1.255	8.02	6,700		14,000		20,700	
00-0072 Rep	DF-20 Rep		8.02	6,900	3	14,000	0	20,900	1
00-0073	DF-21	1.255	8.02	6,800		13,200		20,000	
00-0073 Rep	DF-21 Rep		8.02	6,900	1	13,200	0	20,100	0
00-0074	Wash Composite	1.018	4.95	1,000		2,400		3,400	
00-0074 Rep	Wash Composite (Rep)		4.95	1,000	0	2,300	4	3,300	3
00-0074 Dup	Wash Composite Duplicate	1.025	4.91	1,000		2,400		3,400	
00-0074 Dup Rep	Wash Composite Duplicate (Rep)		4.91	1,100	10	2,500	4	3,600	6
99-2350 MS	*MS Recovery*		*n/a*	*101*		*97*		*100*	
[1] Density calculated from Hot Cell dilutions data. Mass of liquid / Volume of liquid from calibrated pipet.									

Lab Number	Sample ID	TIC		TOC		TC	
		ug/g	RPD(%)	ug/g	RPD(%)	ug/g	RPD(%)
Solids							
00-0083	Wahsed Solids	44,600		1,100		45,700	
00-0083 Dup	Washed Solids Duplicate	57,100	25	1,800	n/a	58,900	25
00-0083 MS	Washed Solids MS Recovery	80%		89%		85%	
00-0083 Rep	Washed Solids (Rep)	51,300		1,600		52,900	
00-0083 Dup Rep	Washed Solids Duplicate (Rep)	38,000	30	2,000	n/a	40,000	28

RPD = Relative Percent Difference (between sample and duplicate/replicate)

The analysis of the subject samples submitted under ASR 5536 was performed by the hot persulfate wet oxidation method. The hot persulfate method uses acid decomposition for TIC and acidic potassium persulfate oxidation at 92-95°C for TOC, all on the same sample, with TC being the sum of the TIC and TOC.

The table above shows the results, rounded to two to three significant figures. The raw data bench sheets and calculation work sheets showing all calculations are attached. All sample results are corrected for average percent recovery of system calibration standards and are also corrected for contribution from the instrument calibration blanks. Per the ASR request, all liquid samples were processed by weight and reported in µg/g. The liquid samples were diluted in the Shielded Analytical Laboratory hot cells to reduce the dose associated with the samples analyzed for TIC/TOC; the hot cell dilution factors are provided in the table. An estimate of the density of each liquid sample was obtained during the dilution process by weighing the volume fraction of the sample used for dilution. The density of each liquid is reported in µg/ml.

Q.C. Comments:

The TIC standard is calcium carbonate and TOC standard is α-Glucose (the certificates of purity are attached). The standard materials were used in solid form for system calibration standards as well as matrix spikes. TIC and TOC percent recovery are determined using the appropriate standard (i.e., calcium carbonate for TIC or glucose for TOC).

The QC for the methods involves calibration blanks, system calibration standards, sample duplicates, and one matrix spike per matrix type.

Calibration Standards: The QC system calibration standards for the 12/09/99 and 12/10/99 analysis runs were all within acceptance criteria, with the average recoveries being 100.2% and 101.1% for TIC and 99.7% and 100.5% for TOC, respectively.

Calibration Blanks: The six calibration blanks run at the beginning and end of the analysis runs were acceptable. The standard deviation calculated from the calibration blanks is less than the estimated method detection limit for both TIC and TOC.

Duplicates: The relative percent differences (RPD) between liquid replicates and the Wash Composite duplicates (00-0074) are within the acceptance criteria of 20%. The RPD for the Washed Solids (00-0083) TIC was above the acceptance criteria. The Washed Solids were analyzed in duplicate twice, with both sets of analyses providing a poor RPD. The TIC concentration for the Washed Solids is quite high, requiring very small sample sizes to be used. The poor reproducibility is most likely due to heterogeneity of the Washed Solids sample combined with the small sample sizes used.

Matrix Spike: The accuracy of the carbon measurements can be estimated by the recovery results from the matrix spike. No matrix spike was prepared for the liquid samples from this ASR. However, a liquid matrix spike prepared from a sample from other ASRs and run in the same batch demonstrated recoveries of 101% for TIC and 97% for TOC, well within the 75% to 125% recovery acceptance criteria. The matrix spike for the solids sample run (i.e., 00-0083, Washed Solids MS) recovered at 80.1% for TIC and 89.2% for TOC. The recoveries are within the acceptance criteria of 75% to 125%. The poorer than normal spike recoveries are most likely attributed to the poor RPDs and sample heterogeneity.

General Comments:

- The reported "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
- Routine precision and bias are typically ±15% or better for non-complex samples that are free of interferences.
- The estimated quantitation limit (EQL) is defined as 5 times the MDL. Results less than 5 times the MDL have higher uncertainties, and RPDs are not calculated for any results less than 5 times the MDL.
- Some results may be reported as less than ("<") values. These less than values represent the sample MDL (method detection limit), which is the system MDL adjusted for the volume of sample used for the analysis. The system MDL is based on the attached pooled historical blank data. The evaluation and calculation of the system MDL is included in the data package.

Report Prepared by: MW Urie **Date** 1-12-00

Review/Approval by: D. Baldwin **Date** 1-13-00

Archive Information:

Files: ASR 5536 Hallen.doc	ASR 5478 5536 5571 Liq+Solids.xls

PNNL Radiochemical Processing Group: TOC/TIC/TC Calculations **Review** Report - Hot Persulfate Method PNL-ALO-381

Client:	**R Hallen**		Analyzer M&TE:	WA92040 -- 701
Project :	**29953**		Balance M&TE:	360-06-01-023
Work Pkg:	**W45525**	**(CMC K88409)**	TOC STD: Glucose CSM-53219>>>	40.00% Carbon <<[G]
Analyzed:	**12/10/99**		TIC STD: CaCO3 CMS-139285>>>	11.99% Carbon <<[C]
ASR:	**5536 (Solids)**			

		Raw TIC (ug C)	Raw TOC (ug C)
Blanks:	Calibration blank (start of batch)	13.8	57.6
	Calibration blank (start of batch)	14.0	52.6
	Calibration blank (end of batch)	14.6	42.1

TIC	TOC	
14.1	50.8	<<< Average (ug C)
0.4	7.9	<<< Std Dev (ug C)
2.16	5.8	<<< Pooled Std Dev (ug C)
6.5	17.3	<<< Method Det. Limit (ug C)

		Total Inorganic Carbon (TIC)					Total Organic Carbon (TOC)			
		[A] Raw TIC (ug)	[B] Blk (ug)	[D] Std wt (g)	TIC % Rec		[E] Raw TOC (ug)	[F] Blk (ug)	[H] Std wt (g)	TOC % Rec
Standards:	Calibration Standard (start of batch)	1070	14	0.00880	100.1		1380	51	0.00330	100.7
	Calibration Standard (start of batch)	1210	14	0.01000	99.7		1082	51	0.00260	99.2
	Calibration blank (end of batch)	969	14	0.00770	103.4		1150	51	0.00270	101.8
		[L] Average TIC % Rec >>>>			101.1	<<[L]	[P] Average TOC % Rec >>>>			100.5

Formulas:

Standard TIC % Recovery = $((A-B)/((C/100)*D))*10^{-6}*100$

Standard TOC % Recovery = $((E-F)/((G/100)*H))*10^{-6}*100$

Sample TIC (ug C/g) = (I-J)/(K*L/100)

Sample TOC (ug C/g = (M-N)/(O*P/100)

Matrix Spike Recoveries:

TIC % Recovery = (((Q-R)/(L/100))-S*T)*100/U

TOC % Recovery = (((Q-R)/(P/100))-S*T)*100/U

TC % Recovery = $((((Q^{TIC}-R^{TIC})/(L/100))-V^{TIC})+(((Q^{TOC}-R^{TOC})/(P/100))-V^{TOC}))*100/U^{TIC+TOC}$

Comments:

Due to the precision carried in the spreadsheet, some results may appear to be slightly off due to rounding.
The Pooled SD is the averaged SD for a recent list of 12 sample batches. MDL is based upon the Pooled SD. MDL = 3 x pooled SD.
If either the Sample or Duplicate are < 5x mdl, then the RPD is not calculated and displayed as "n/a".
TIC and TOC are measured; TC is the sum of the TIC and TOC results.

PNNL Radiochemical Processing Group: TOC/TIC/TC Calculations **Review** Report - Hot Persulfate Method PNL-ALO-381

Client: R Hallen
Project : 29953
Work Pkg: W45525 (CMC K88409)
Analyzed: 12/10/99
ASR: 5536 (Solids)

Analyzer M&TE: WA92040 -- 701
Balance M&TE: 360-06-01-023
TOC STD: Glucose CSM-53219>>> 40.00% Carbon <<[G]
TIC STD: $CaCO_3$ CMS-139285>>> 11.99% Carbon <<[C]

Sample Results

ACL Number	Client Sample ID	[I] Raw TIC (ug C)	[J] Blk (ug C)	[K] Sam Wt(g)	TIC (ug C/g)	TIC RPD (%)	[M] Raw TOC (ug C)	[N] Blk (ug C)	[O] Sam Wt(g)	TOC (ug C/g)	TOC RPD (%)	TC (ug C/g)	TC RPD (%)
00-0083	Wahsed Solids	1280	14	0.0281	44,568		81	51	0.0281	1,070		45,638	
00-0083 Dup	Washed Solids Duplicate	1700	14	0.0292	57,119	25	104	51	0.0292	1,813	n/a	58,932	25
00-0083 MS	Washed Solids MS	1982	14	0.0286	see below		835	51	0.0286	see below		see below	
00-0083 Rep	Washed Solids (Rep)	1600	14	0.0306	51,273		100	51	0.0306	1,600		52,873	
00-0083 Dup R	Washed Solids Duplicate (Rep)	1332	14	0.0343	38,012	30	120	51	0.0343	2,008	n/a	40,019	28

Matrix Spike Results

ACL Number	Client Sample ID	[Q] Raw MS (ug C)	[R] MS Blk (ug C)	[S] Sam (ug C/g)	[T] MS Sam Wt(g)	[V] Sample (ug C)	Spike wt (g)	[U] Spike (ug C)	MS % Recovery	
00-0083 MS	TIC Recovery	1982	14	44568	0.0286	1275	0.0070	839	80.1	TIC
	TOC Recovery	835	51	1070	0.0286	31	0.0021	840	89.2	TOC
	Total Carbon Recovery (TIC + TOC)							1679	84.7	TC

Preparer/date: MW Urie 12-17-99

Reviewer/date: JT Baldwin 1-13-00

WT% SOLIDS DATA SHEET
(325 SHIELDED ANALYTICAL LABORATORY)

CLIENT: RT HALLEN   WORK PACKAGE: W45525   ASR/ARF/LOI/TI: ASR5536

QA PLAN: MCS-033   IMPACT LEVEL: ______   PROCEDURE NUMBER: PNL-ALO-504

RT HALLEN
Date ·/4/00
~~Route~~
File 11-52
Copy record - RT Hallen

00-0083 WASHED SOLIDS
SAMPLE IDENTIFICATION

ACL NUMBER	CLIENT IDENTIFICATION	TARE WEIGHT (G)	(A) SAMPLE WET WEIGHT PLUS TARE	(B) SAMPLE DRY WEIGHT PLUS TARE		WEIGHT % SOLIDS
00-0083	WASHED SOLIDS	16.7572	21.6748	18.5767	18.5685 18.5696	36.83 %
00-0083-DUP	WASHED SOLIDS	17.0414	23.4848	19.3895	19.3819 19.3840	36.32 %

$$WT\%\ SOLIDS = \frac{B - TARE}{A - TARE} \times 100$$

DATE/TIME IN: 10-21-99 1100   OVEN TEMPERATURE: 107 °C

DATE/TIME OUT: 10-22-99 1000   OVEN TEMPERATURE: 104 °C

BALANCE: CELL 2 (360-06-01-016) ✓

THERMOCOUPLE: 2116   BALANCE: CELL 5 (360-06-01-039) ____

Analyst: [signature]   Date: 10-21-99   Reviewer: [signature]   Date: 11/1/99

APPENDIX C: RHEOGRAMS

Figure 1. 50 cP Standard , Brookfield Lot 102298, Analyzed on Bohlin CS Before AN-107 Initial Diluted Feed Samples 1/22/99

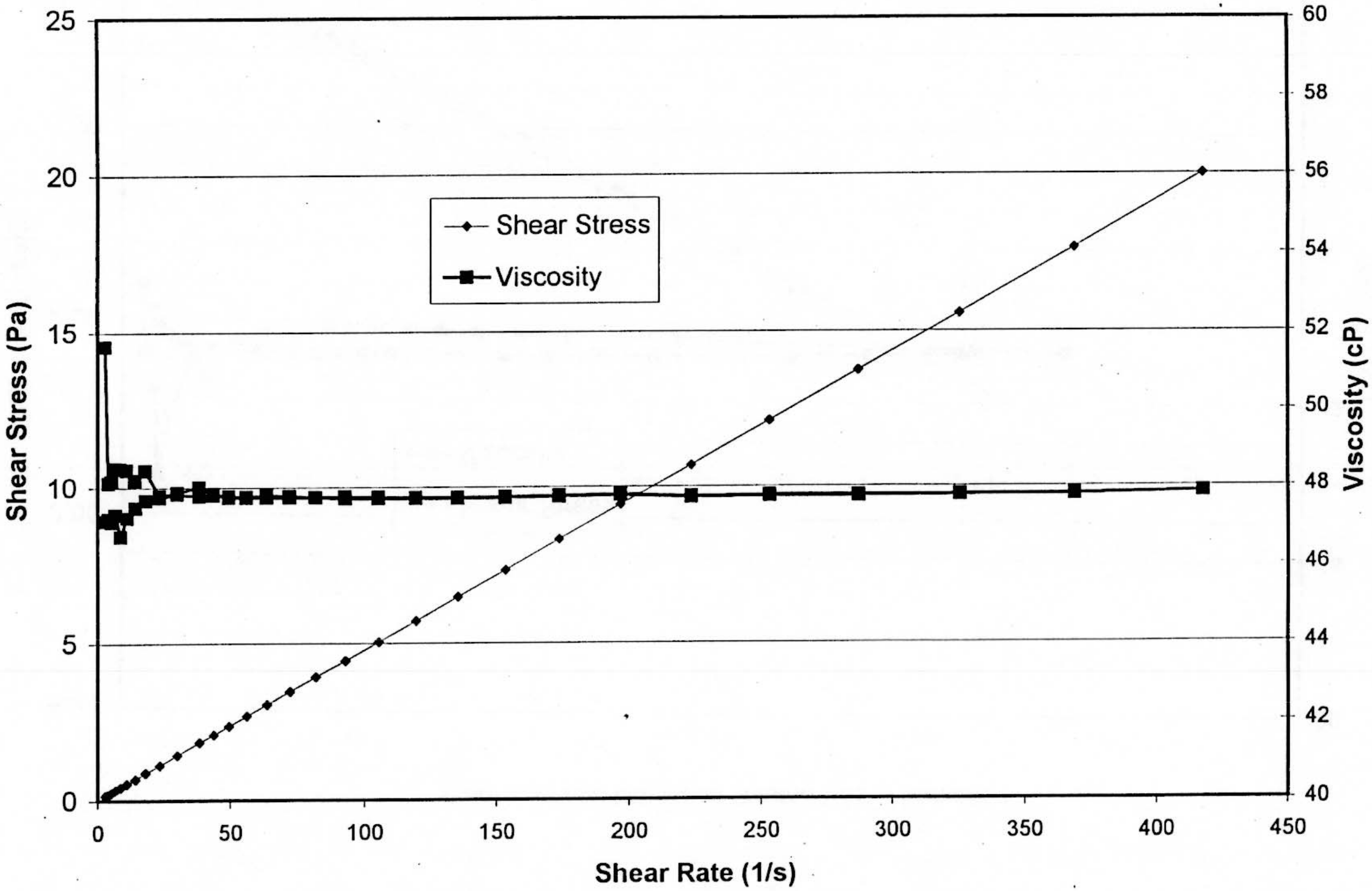


Figure 2. AN-107 Initial Diluted Feed: Sample 1

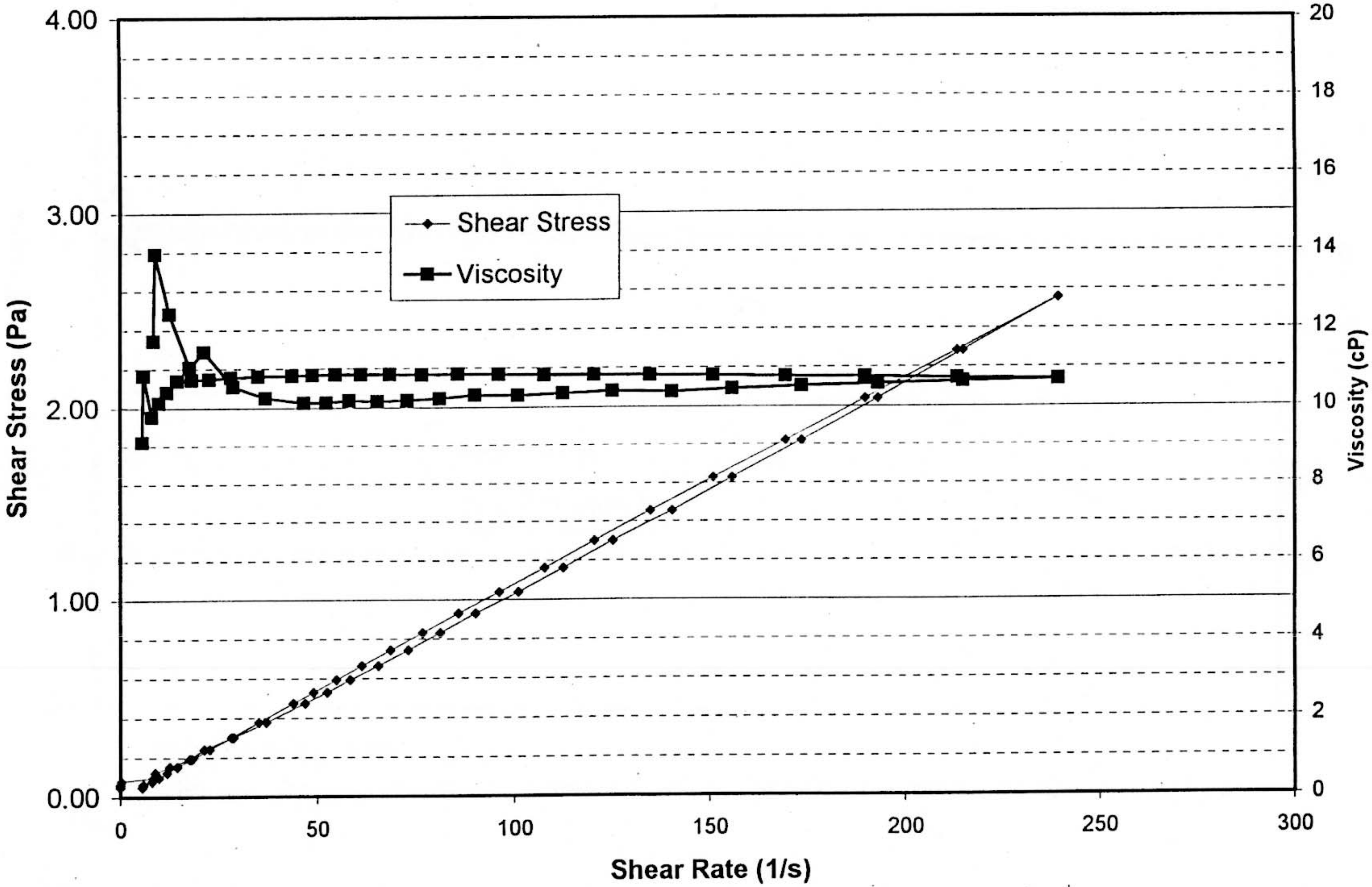


Figure 3. AN-107 Diluted Feed: Sample 2

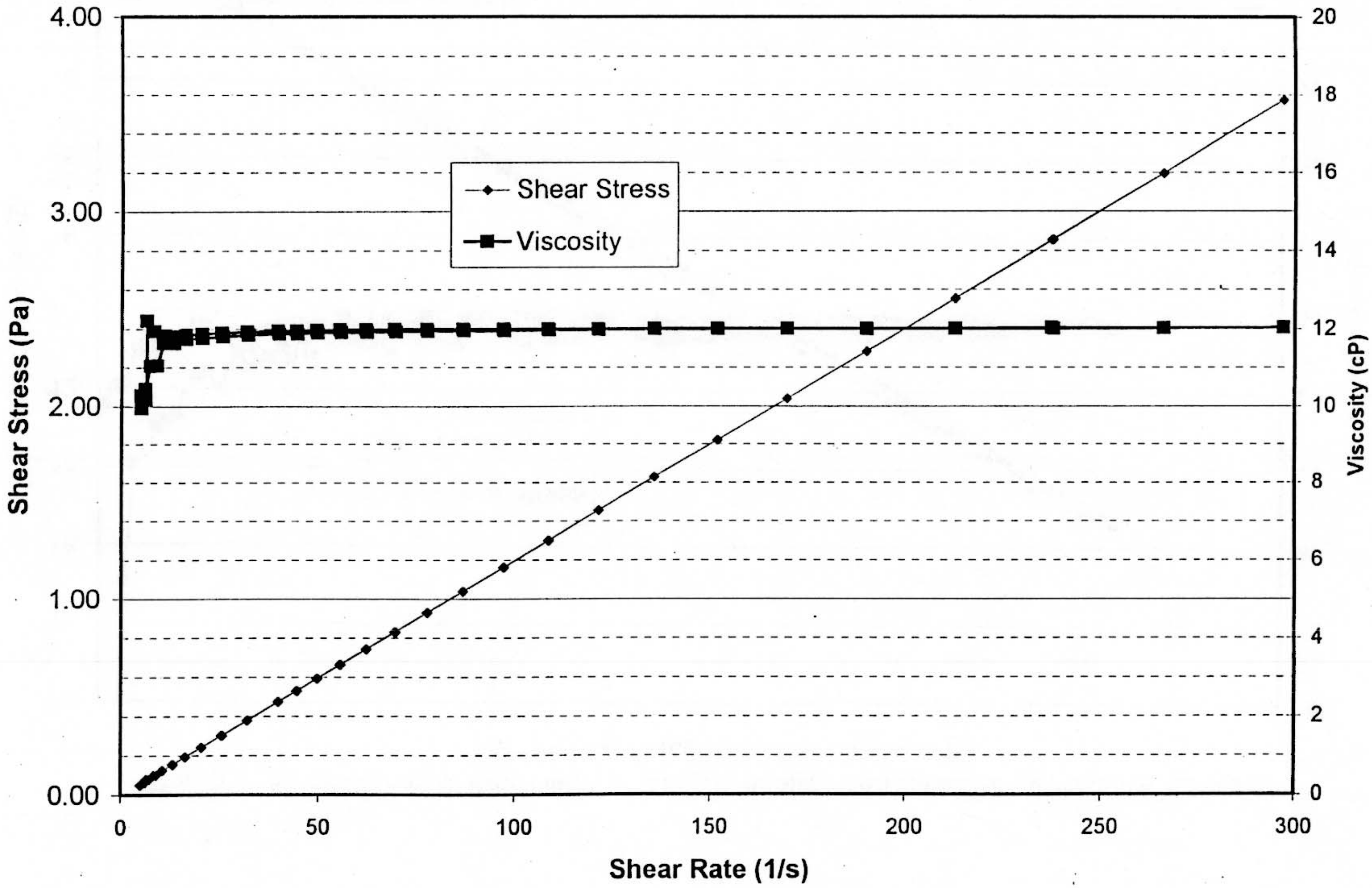


Figure 4. 50 cP Standard, Brookfield Lot 102298, Analyzed on Haake M5 Viscometer Before AN-107 SR/TRU Percipitated Samples 8/24/99

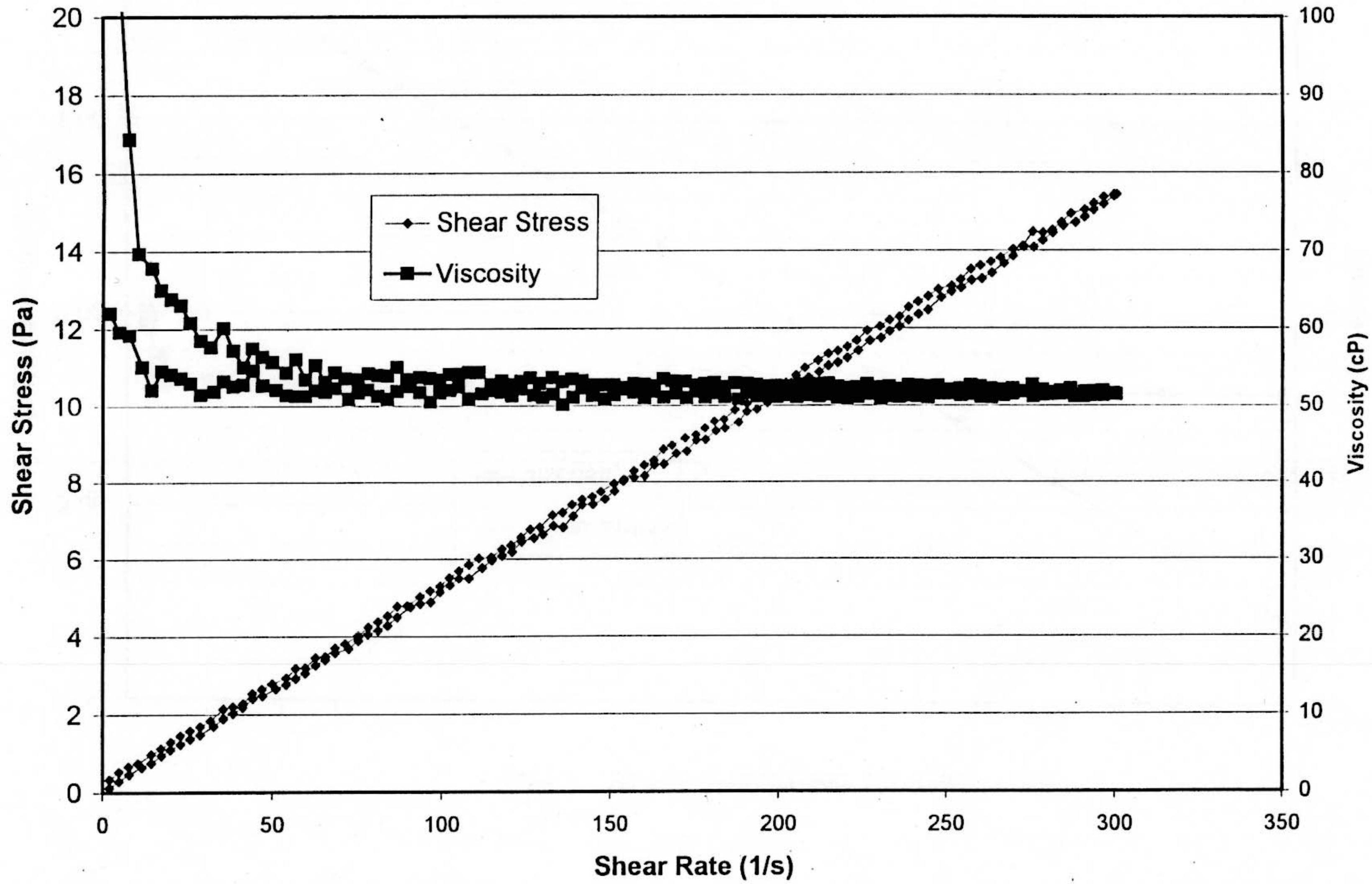


Figure 5. AN-107 Sr/TRU Precipitated Slurry: Sample 1 Analysis 1

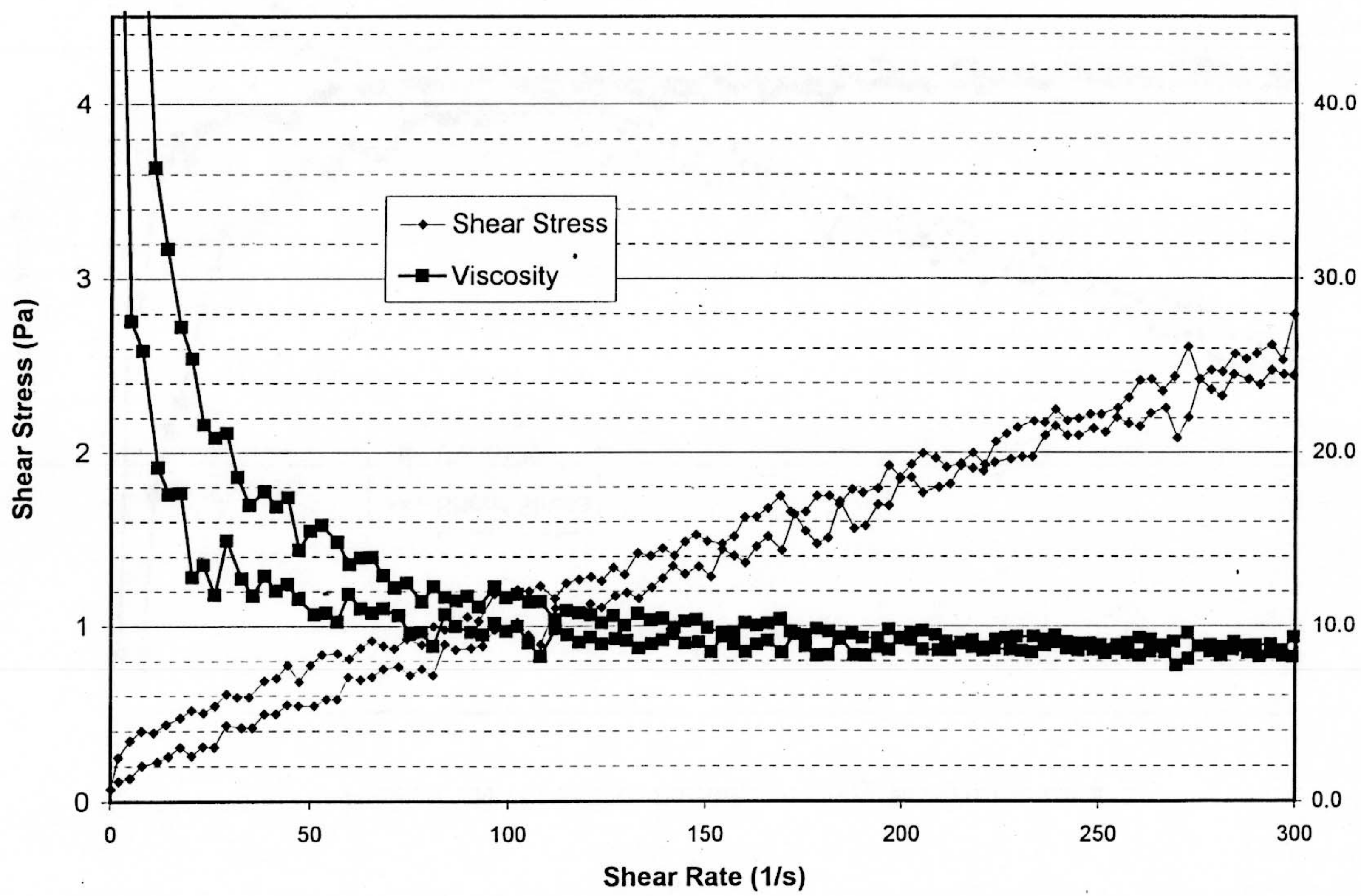


Figure 6. AN-107 Sr/TRU Precipitated Slurry: Sample 1 Analysis 2

Figure 7. AN-107 Sr/TRU Precipitated Slurry: Sample 1 Analysis 3

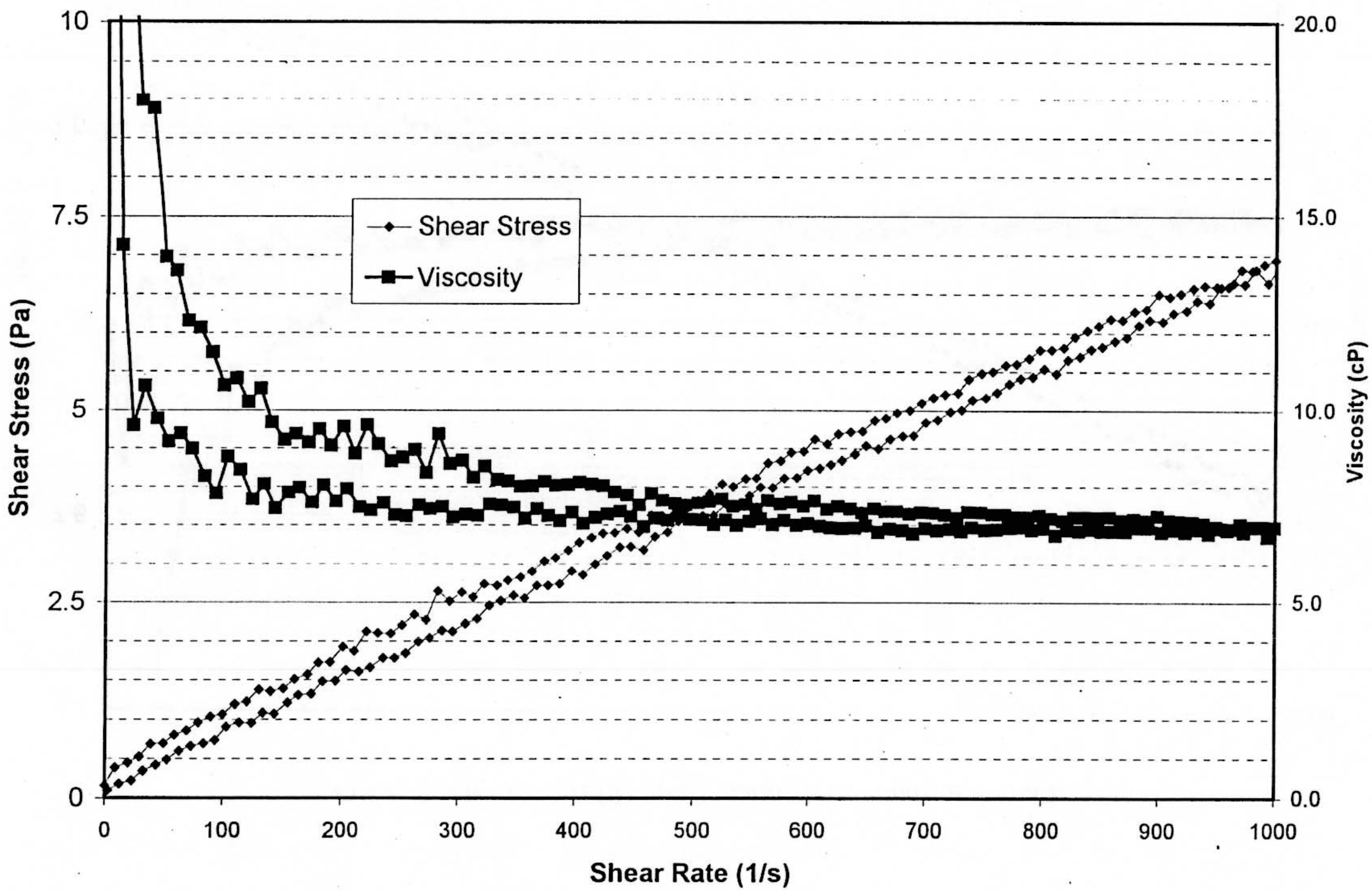


Figure 8. AN-107 Sr/TRU Precipitated Slurry: Sample 2 Analysis 1

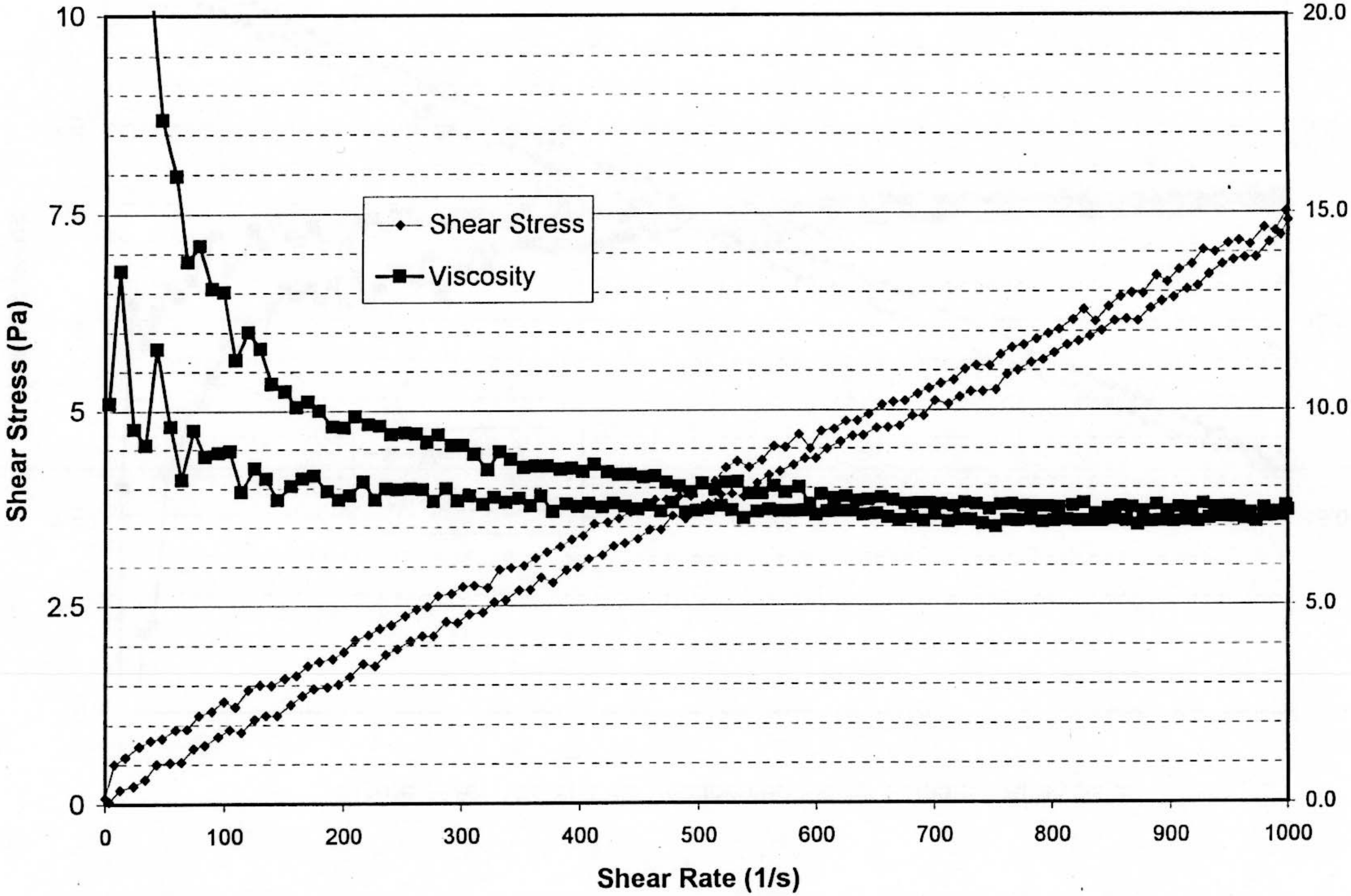


Figure 9. AN-107 Sr/TRU Precipitated Slurry: Sample 2 Analysis 2

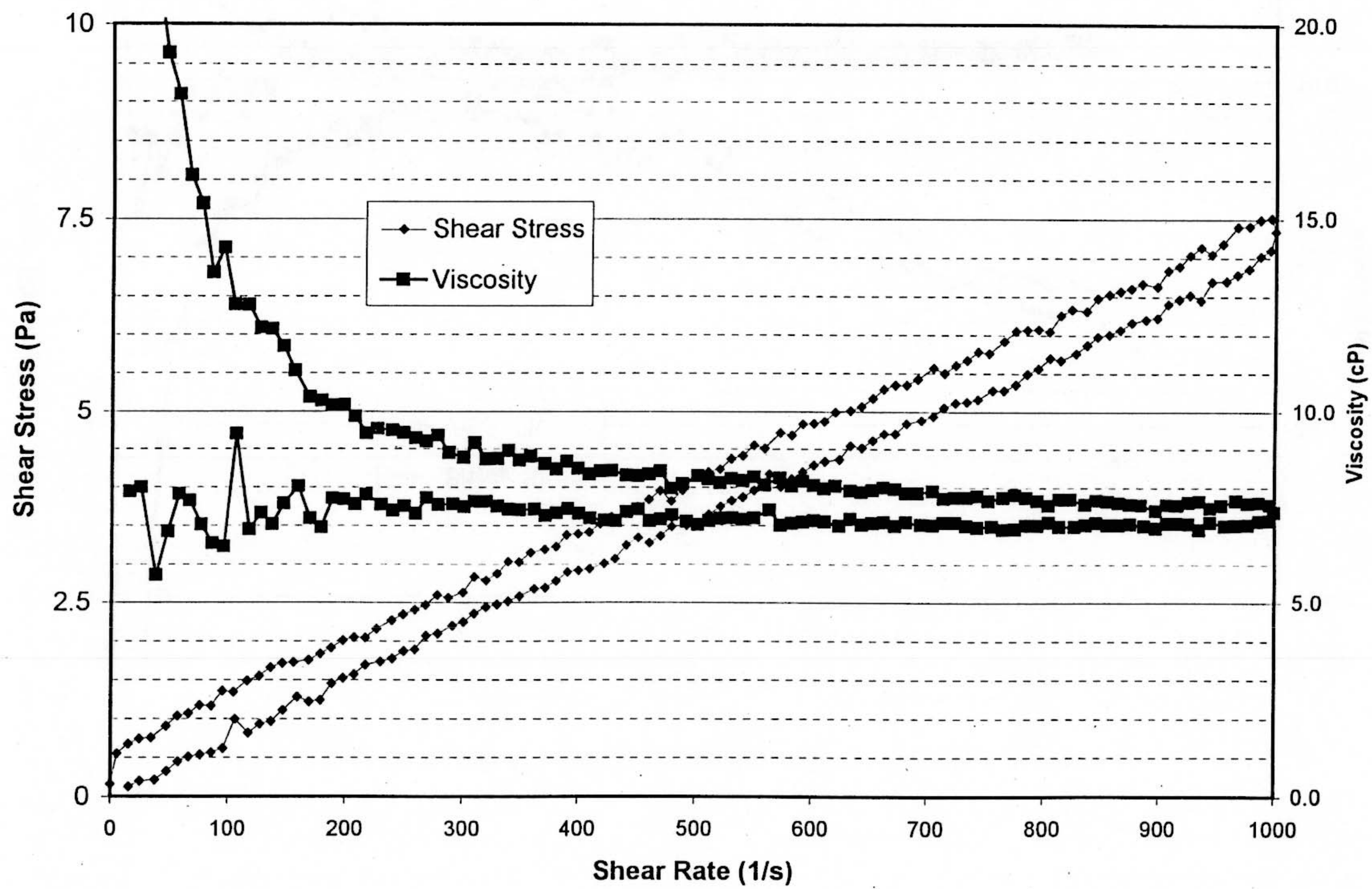
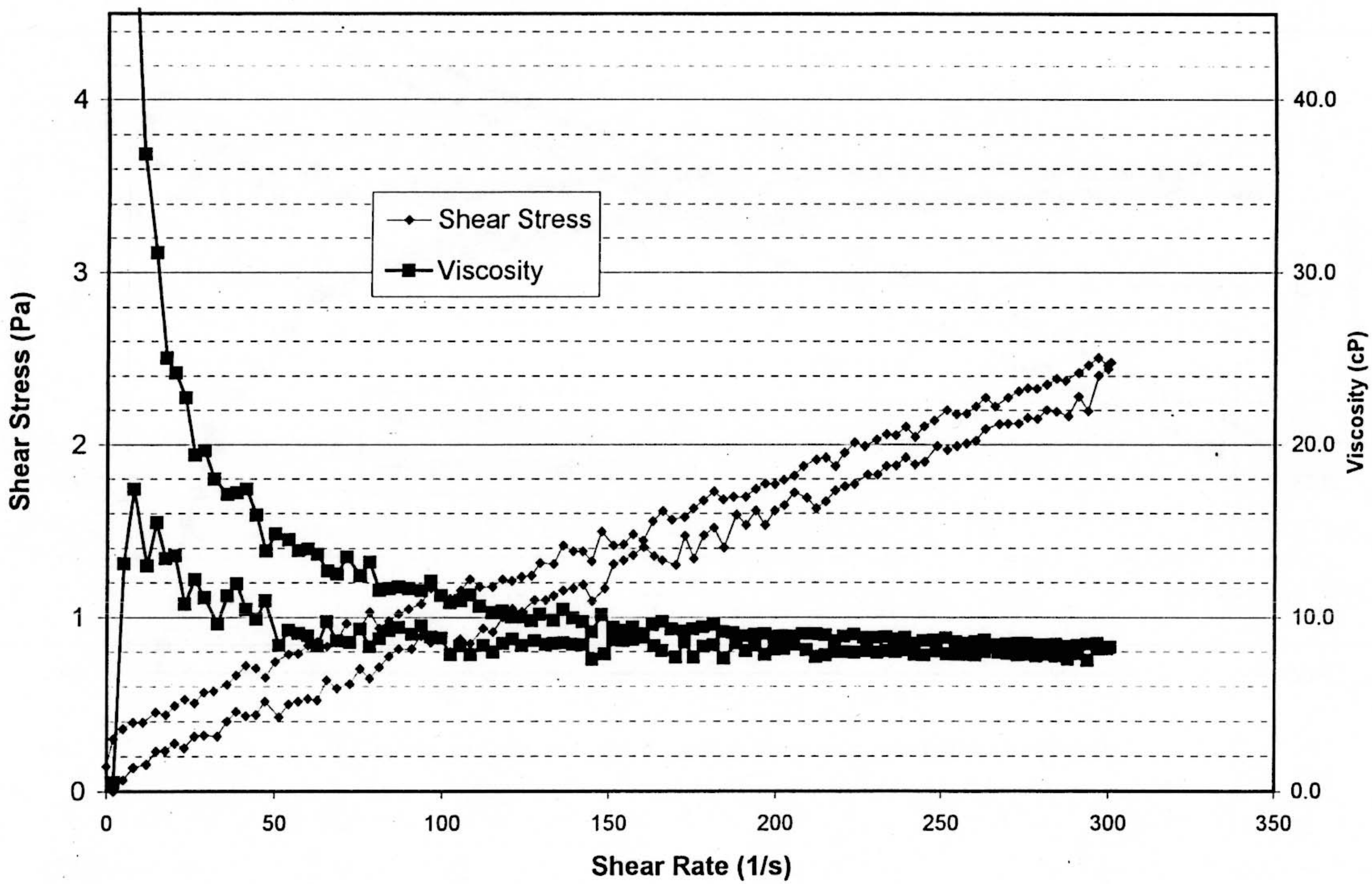


Figure 10. AN-107 Sr/TRU Precipitated Slurry: Sample 2 Analysis 3



APPENDIX D: CALCULATIONS

TI-052, actual test conditions for An-107 DF Sr/TRU removal and CUF tests

		Calculated Mass Dilution Factor			
Weight of starting AN-107 DF	1845.44 grams				
Density from Paul Bredt	1.32 g/mL				
Total volume of treated AN-107D	1398.06 mL				
3.5185M NaOH added	296.548 grams				
Density of NaOH	1.12261 g/mL				
Volume of NaOH added	264.16 mL				
		1.1607			
1.0M Sr added	165.367 grams				
Density of Sr	1.15852 g/mL				
Volume of Sr added	142.74 mL				
1.0M MnO4 added	105.558 grams				
Density of MnO4r	1.09432 g/mL				
Volume of MnO4 added	96.46 mL				
		1.3075	samples DF-01 to 04	total mass	total volume
Density of resulting slurry	1.2975 g/mL			2412.911	1859.6617
Density of CUF Filtrate	1.2855 g/mL		sample DF-11		

Waste was further dilute, approximately 25% and rerun through the CUF

grams CUF slurry,	837.58 grams			
grams filtrate,	1189.89 grams			
plus DF-07,08,09,10	85 grams			
	2112.47 grams			
diluted with	406.51 grams of first CUF was	1.1924	use value of 1.19	

Calculated

	volume	[Na]	moles of N	new volum	new [Na]	[Sr]	[MnO4]	[HO-]
initial waste	1.4	7.5	10.5					0.7
water	0.2156	0	0	1.6156	6.499133			
19M NaOH	0.049	19	0.931	1.6646	6.867115			
1M Sr	0.1428	0	0	1.8074	6.324555			
1M MnO4	0.0966	1	0.0966	1.904	6.054412	0.075	0.050735	1.003676

Actual:

	volume	[Na]	moles of N	new volum	new [Na]	[Sr]	[MnO4]	[HO-]
initial waste	1.398059	8	11.184473					0.7
water	0	0	0	1.398059	8			
3.51M NaOH	0.26416	3.5185	0.929447	1.662219	7.2878			
1M Sr	0.14274	0	0	1.804959	6.711465			
1M MnO4	0.09646	1.001	0.0965565	1.901419	6.42177	0.074995	0.050781	1.003508

Total volume of treated AN-107DF	1398.0591	mL	
3.5185M NaOH added	296.548	grams	
Density of NaOH	1.1226075	g/mL	
Volume of NaOH added	264.16	mL	
			1.160692
1.0M Sr added	165.367	grams	
Density of Sr	1.158519	g/mL	
Volume of Sr added	142.74	mL	
1.0M MnO4 added	105.558	grams	
Density of MnO4r	1.0943189	g/mL	
Volume of MnO4 added	96.46	mL	
			1.3075 samples DF-01 to 04
Density of resulting slurry	1.2975	g/mL	
Density of CUF Filtrate	1.2855	g/mL	sample DF-11

Waste was further dilute, approximately 25% and rerun through the CUF

grams CUF slurry,	837.58	grams
grams filtrate,	1189.89	grams
plus DF-07,08,09,10	85	grams
	2112.47	grams

diluted with 406.51 grams of first CUF was 1.192434 use value of 1.19

		Total	Supernatant	Centrifuged Wet Solids											
Jar/Bottle ID (1)	Lab ID	Mass (g) (2)	Mass (g)	Wt%	Mass (g)	Wt%									
AN-107 CA	99-0645/0647	81.81	77.61	94.90%	4.2	5.10%									
Jar/Bottle ID (1)	Lab ID		Density (g/mL)												
	Sample	Primary	Duplicate	Average											
AN-107 CA	99-0645	1.368	1.36	1.36											
Tank Material	AN 107 Diluted Feed (1)														
Matrix	Supernatant				Centrifuged Wet Solids										
Dissolution	Acid Digest														
					KOH-KNO3 Fusion				Acid Digest (2)						
Lab ID	AVERAGE	99-0645	99-0645-d		AVERAG	99-0647	99-0647-d		AVERAGE	99-0647	99-0647-d		AVERAGE		recombined
Sample ID		Sample	Duplicate	RPD		Sample	Duplicate	RPD		Sample	Duplicate	RPD	SOLIDS		waste
Units	g/mL	g/mL	g/mL	(%)	g/g	g/g	g/g	(%)	g/g	g/g	g/g	(%)			ug/g
BNFL List															
Ag		< 3	< 3			< 23	< 25			< 3	< 3				0
Al	3930	4,040	3,820	6	7500	7,550	7,450	1	7395	7,140	7,650	7	7447.5		3122.15338
Ba	4.1	[4.1]	[4.0]			[44]	[44]		45.5	45	46	2	45.5		5.18145588
Ca	438.5	461	416	10		[780]	[520]		368	359	377	5	368		324.750721
Cd	47	48	46	5		[37]	[36]		33.5	33	34	3	33.5		34.5048235
Co		< 4	< 4			< 46	< 51			< 6	< 7				0
Cr	145.5	149	142	5	721.5	725	718	1	710	697	723	4	715.75		138.032294
Cu	21	[22]	[20]			< 23	< 25		19.5	[19]	[20]		19.5		15.6481765
Fe	1140	1,170	1,110	5	9315	9,960	8,670	14	8475	8,260	8,690	5	8895		1249.13029
K	1270	[1,300]	[1,240]			n/a	n/a		658.5	[670]	[647]		658.5		1239
La	23	[23]	[22]			[65]	[60]		110	108	112	4	110		21.6592647
Mg		< 12	< 12			< 93	< 102		30	[30]	[30]		30		1.53
Mn	107	108	106	2	5020	4,910	5,130	4	4954.5	4,920	4,989	1	4987.25		329.013721
Mo		< 6	< 6			< 46	< 51			< 6	< 7				0
Na	173500	176,000	171,000	3	136500	134,000	139,000	4	136500	134,000	139,000	4	136500		128028.779
Ni	392	402	382	5		n/a	n/a		277	269	285	6	277		287.662294
Pb	256	263	249	5		[580]	[640]		769.5	755	784	4	769.5		217.879794
Si		< 45	< 44			< 463	< 509			[264]	[419]				0
Ti		< 3	< 3			< 23	< 25			[4.5]	[4.8]				0
U		< 244	< 241			< 1,851	< 2,036			< 231	< 279				0
Zn	19	[22]	[16]			< 46	< 51		64	64	[67]		64		16.5220882
Zr	43	[42]	[44]			[110]	[92]		201.5	197	206	4	201.5		40.2816471
Other Analytes Detected															
As	95.5	[98]	[93]			< 231	< 254		80	[77]	[83]		80		70.7193382
Ce	27	[27]	[26]			[190]	< 204		215.5	[212]	[219]		215.5		29.8309412
Nd	70.5	[73]	[68]			[210]	[190]		318.5	311	326	5	318.5		65.4379853
P	496.5	505	488	3		[500]	[520]		427	418	436	4	427		368.231779
Sr	2.6	[2.7]	[2.5]			< 14	< 15		6.3	[6.2]	[6.3]		6.3		2.79
Y	11	[11]	[11]			< 46	< 51		31.5	[31]	[32]		31.5		9.28223529

(1) Overall error for reported results is estimated to be within 15%; however results in brackets "[ ]" are less than the estimated quantitation level (i.e., 10-times MDL listed in Table 5.1) and error is anticipated to be greater than 15%..

(2) Solids acid digestion results normalized to KOH-KNO3 fusion sodium results. See narrative 4.1.

(3) RPD only calculated when both sample and duplicate exceed estimated quantitation level.

Tank Material	AN-107 Diluted Feed														
Matrix	Supernatant							Centrifuged Wet Solids (1)							
Dissolution	Acid Digest							KOH-KNO3 Fusion							
Sample ID			99-0645		99-0645-d					99-0647		99-0647-d		RPD	Recombined
Units (%Error)	AVERAGE	Average in	Sample		Duplicate		RPD	AVERAGE		Sample		Duplicate		(%)	Waste
	Ci/mL %Err	uCi/g	Ci/mL %Er	% Error	Ci/mL	% Error	(%)	Ci/g %Err		Ci/g %E	% Error	Ci/g	% Error		uCi/g
Co-60 (GEA) (3)	1.13E-01	8.56E-02	1.11E-01	7	1.15E-01	7	4			< 9.E-2		< 9.E-2			7.89E-02
Sr-90	7.59E+01	5.75E+01	7.72E+01	5	7.46E+01	5	4	1.92E+02		1.90E+02	4	1.93E+02	4	2	6.27E+01
Cs-134 (GEA) (3)		0.00E+00	< 3.E-2		< 3.E-2					< 8.E-2		< 9.E-2			
Cs-137 (GEA)	2.56E+02	1.94E+02	2.61E+02	2	2.50E+02	2	4	1.65E+02		1.65E+02	2	1.65E+02	2	0	1.87E+02
Eu-154 (GEA)	6.12E-01	4.63E-01	6.20E-01	4	6.03E-01	4	3	1.31E+00		1.44E+00	5	1.17E+00	7	21	4.93E-01
Eu-155 (GEA)	3.56E-01	2.69E-01	4.50E-01	16	2.61E-01	25		8.18E-01		9.87E-01	14	6.49E-01	21		2.90E-01
Pu-238 (3)	7.69E-03	5.83E-03	7.02E-03	15	8.36E-03	9		4.38E-02		5.01E-02	7	3.75E-02	9	29	7.60E-03
Pu-239+Pu-240 (3)	3.14E-02	2.38E-02	3.08E-02	7	3.20E-02	5	4	1.51E-01		1.68E-01	5	1.33E-01	5	23	2.96E-02
Am-241 (GEA)	3.98E-01	3.02E-01	5.66E-01	23	2.30E-01	50		2.09E+00		2.49E+00	12	1.68E+00	18		3.84E-01
Am-241 (AEA)	3.79E-01	2.87E-01	3.93E-01	5	3.64E-01	5	8	1.47E+00		1.67E+00	5	1.27E+00	5	4	3.39E-01
Cm-242 (3)	1.44E-03	1.09E-03	1.60E-03	21	1.28E-03	28		4.21E-03		4.18E-03	16	4.24E-03	11		1.22E-03
Cm-243+Cm-244	1.23E-02	9.28E-03	1.19E-02	9	1.26E-02	10	6	3.10E-02		3.67E-02	7	2.52E-02	23		1.01E-02
		0.00E+00													
Total Beta	4.50E+02	3.41E+02	4.66E+02	3	4.34E+02	3	7	5.15E+02		5.20E+02	4	5.09E+02	4	2	3.40E+02
Total Alpha	4.47E-01	3.38E-01	4.43E-01	3	4.50E-01	3	2	1.83E+00		2.14E+00	4	1.52E+00	5	34	4.05E-01
Alpha Sum	4.32E-01	3.27E-01	4.45E-01	4	4.19E-01	4	6	1.70E+00		1.93E+00	4	1.47E+00	4	27	3.88E-01
		0.00E+00	g/mL %Err		g/g %	g/g %Err	g/g %Err								
(1s)		0.00E+00													ug/g
Total Cs (3)	1.20E+01	9.09E+00	1.23E+01	2	1.17E+01	2	4	7.68E+00		7.72E+00	2%	7.63E+00	2	1	8.76E+00
Total U	7.31E+01	5.54E+01	7.44E+01	2	7.18E+01	2	4	1.03E+02		1.03E+02	2%	1.03E+02	2	0	5.63E+01

(1) Total cesium performed by TIMS from water leach of solids, not from KOH-KNO3 fusion.

(2) RPD only calculated when both sample and duplicate have error uncertainties <10%.

Tank Material	AN-107 Diluted Feed														
Matrix	Supernatant					Centrifuged Wet Solids									
Lab ID			99-0645	99-0645-d				99-0647	99-0647-d						
Sample ID		AVERAGE	Sample	Duplicate	RPD		AVERAG	Sample	Duplicate	RPD					
Units	Type of Prep	g/mL (2)	g/mL (2)	g/mL (2)	(%)	Type of Prep	g/g (2)	g/g (2)	g/g (2)	(%)	ug/g		ug/g	MW	conc, M
Tc-99 (ICP/MS)	Acid Digest	4.315	4.4	4.23	4	Fusion	3.77	3.62	3.92	8	3.203252	Tc-99 (ICP	2.76E+00		
Tc-99 (ICP/MS)	Direct/Dilution	4.035	4.09	3.98	3	n/a		n/a	n/a			Tc-99 (ICP	0.00E+00		
TIC	Direct/Dilution	16300	16,400	16,200	1	Direct	17850	18,200	17,500	4	12284.39	TIC	1.06E+04	12	1.16E+00
TOC	Direct/Dilution	29900	30,000	29,800	0	Direct	32000	31,100	32,900	1	22496.04	TOC	1.94E+04	12	2.13E+00
TC (sum)	Direct/Dilution	46200	46,400	46,000	1	Direct	50800	51,200	50,400	2	34828.89	TC (sum)	3.00E+04	12	3.30E+00
Fluoride	Direct/Dilution	6350	6,300	6,400	2	Water Leach	4400	4,500	4,300	3	4655.393	Fluoride	4.01E+03	19	2.79E-01
Chloride	Direct/Dilution	1400	1,400	1,400	2	Water Leach		<1200	<1200		976.9118	Chloride	8.42E+02		
Nitrite (3)	Direct/Dilution	51350	51,100	51,600	1	Water Leach	31050	30,900	31,200	1	37415.28	Nitrite (3)	3.22E+04	46	9.25E-01
Bromide (3)	Direct/Dilution	#DIV/0!	<490	<480		Water Leach	#DIV/0!	<1200	<1200			Bromide (	0.00E+00		
Nitrate	Direct/Dilution	161000	161,000	161,000	0	Water Leach	111000	111,000	111,000	0	118005.9	Nitrate	1.02E+05	62	2.16E+00
Phosphate	Direct/Dilution	3000	3,000	3,000	2	Water Leach		<2400	<2400		2093.382	Phosphate	1.80E+03	31	7.68E-02
Sulfate	Direct/Dilution	7650	7,900	7,400	6	Water Leach	7000	7,000	7,000	1	5695.125	Sulfate	4.91E+03	96	6.75E-02
Oxalate (3)	Direct/Dilution	1300	1,300	1,300	1	Water Leach	32100	32,200	32,000	1	2544.232	Oxalate (3	2.19E+03		
		mmole/mL	mmole/mL	mmole/mL			mmole/mL	mmole/mL	mmole/mL		mmole/g		mmole/g ??		
Hydroxide	Direct/Dilution	0.717	0.722	0.712	1	n/m					0.500318	Hydroxide	0.8650		1.14176035
		pH	pH	pH			pH	pH	pH						
pH	Direct	n/m	13.33	13.31	0	n/m	n/m	n/m	n/m						
(1) RPD only calculated when sample and duplicate results above threshold for method's RPD calculation. (Calculated prior to rounding)															
(2) Value in parentheses for Tc-99 are in units of Ci/mL for supernatants and Ci/g for solids.															
(3) Not an analyte of interest specified in Test Plan BNFL-29953-6; included for information only.															
For the Sr/TRU precipitation,		1845.438	grams of waste was diluted with			296.548	grams of 3.5185 M NaOH		264.16	mL	0.929447	moles of sodium and OH added			
											21.36777	grams of sodium			
	so all components diluted by		0.86155465	except sodium and hydroxide											

Recombined Waste	uCi/g	Mass Dilution	Concentration After Treatment DF-01	DF-02	DF-03	DF-04	DF-11
Co-60 (GEA) (3)	0.07885						
Sr-90	62.7291	1.3075	0.993	0.412	0.195	0.394	0.585
Cs-134 (GEA) (3)							
Cs-137 (GEA)	186.701						
Eu-154 (GEA)	0.49326						
Eu-155 (GEA)	0.28978						
Pu-238 (3)	0.0076						
Pu-239+Pu-240	0.02959						
Am-241 (GEA)	0.38406						
Am-241 (AEA)	0.33909	1.3075	0.0101	0.00482	0.00403	0.00464	0.00936
Cm-242 (3)	0.00122	1.3075	0.0000386	0.0000242	0.0000189	0.0000222	4.2E-05
Cm-243+Cm-24	0.01013	1.3075	0.000424	0.00021	0.000179	0.0002	0.00039
Total Beta	340.247						
Total Alpha	0.4049						

	Decontamination Factors* DF-01	DF-02	DF-03	DF-04	DF-11
Sr-90	48	116	246	122	82
Am-241 (AEA)	26	54	64	56	28
Cm-242 (3)	24	39	49	42	22
Cm-243+Cm-244	18	37	43	39	20

ALO ID Client ID	Alpha Error %	Sr-90 Error %	Pu-239 + Pu-240 Error %	Pu-238 Error %	Am-241 Error %	Cm-243+ Cm-244 Error %	Cm-242 Error %	*Comment
DF-01		0.946			0.0101	0.00042	4E-05	sampled before heating and filter immediately
DF-01		1.04						
DF-02		0.412			0.00482	0.00021	2E-05	sampled before heating left as slurry until sample prep.
DF-03		0.195			0.00403	0.00018	2E-05	sampled after heating left as slurry until sample prep
DF-04		0.394			0.00464	0.0002	2E-05	duplicate of DF-03
DF-11		0.585			0.00936	0.00039	4E-05	CUF filtrate

Recombined Waste	uCi/g	Mass Dilution	Concentration After Treatment DF-14	DF-20	DF-21	AVE	Supernate only	uCi/g
Co-60 (GEA) (3)	0.07885	1.5559		5.19E-2	4.80E-2	4.99E-2	Co-60 (GE	0.08561
Sr-90	62.7291	1.5559	7.30E-1	4.74E-1	4.81E-1	4.77E-1	Sr-90	57.5
Cs-134 (GEA) (3)							Cs-134 (G	0
Cs-137 (GEA)	186.701	1.5559		1.25E+2	1.22E+2	1.23E+2	Cs-137 (G	193.561
Eu-154 (GEA)	0.49326	1.5559		2.98E-2	2.73E-2	2.85E-2	Eu-154 (G	0.46326
Eu-155 (GEA)	0.28978	1.5559					Eu-155 (G	0.26932
Pu-238 (3)	0.0076	1.5559		1.68E-4	1.66E-4	1.67E-4	Pu-238 (3)	0.00583
Pu-239+Pu-240	0.02959	1.5559		6.46E-4	6.18E-4	6.32E-4	Pu-239+Pu	0.02379
Am-241 (GEA)	0.38406						Am-241 (G	0.30152
Am-241 (AEA)	0.33909	1.5559	3.29E-3	4.89E-3	5.04E-3	4.97E-3	Am-241 (A	0.28674
Cm-242 (3)	0.00122	1.5559	1.53E-5	2.21E-5	2.42E-5	2.32E-5	Cm-242 (3)	0.00109
Cm-243+Cm-24	0.01013	1.5559	1.29E-4	2.20E-4	2.43E-4	2.32E-4	Cm-243+C	0.00928
								0
Total Beta	340.247						Total Beta	340.909
Total Alpha	0.4049	1.5559		8.14E-3	7.72E-3	7.93E-3	Total Alpha	0.33826

Total Sample	Decontamination Factors** DF-14	DF-20	DF-21
Co-60 (GEA) (3)		1	1
Sr-90	55	85	84
Cs-134 (GEA) (3)			
Cs-137 (GEA)		1	1
Eu-154 (GEA)		11	12
Eu-155 (GEA)			
Pu-238 (3)		29	29
Pu-239+Pu-240 (3)		29	31
Am-241 (GEA)			
Am-241 (AEA)	66	45	43
Cm-242 (3)	51	35	32
Cm-243+Cm-244	50	30	27
Total Beta			
Total Alpha		32	34

Supernate	Decontamination Factors** DF-14	DF-20	DF-21
Co-60 (GEA) (3)		1	1
Sr-90	51	78	77
Cs-134 (GEA) (3)			
Cs-137 (GEA)		1	1
Eu-154 (GEA)		10	11
Eu-155 (GEA)			
Pu-238 (3)		22	23
Pu-239+Pu-240 (3)		24	25
Am-241 (GEA)			
Am-241 (AEA)	56	38	37
Cm-242 (3)	46	32	29
Cm-243+Cm-2	46	27	25
Total Beta			
Total Alpha		27	28

Removal of Tc by Sr/TRU ppt

Supernate only	ug/g	Mass Dilution from Sr/TRU Treatment	Concentration After Treatment DF-20 ug/g	DF-20-dup ug/g	DF-21 ug/g
Tc-99	3.172794118				
Tc-99	2.966911765				
Average	3.069852941	1.555925526	2.59	2.56	2.86

Percent Removed			
Tc	-31.27%	-29.75%	-44.96%
	0.761779	0.770706	0.689863

AN-107 Diluted Feed analyses, Report -003

Centrifuged Wet Solids

Type of Prep	AVERAGE	99-0647 Sample	99-0647-d Duplicate	RPD (%)
Fusion	3.77	3.62	3.92	8

Sr/TRU Solids

			ug/g
00-0083	Washed Solids	9b03a10	6.73
00-0083-DUP	Washed Solids	9b03a11	10.5

DF-20 ave	DF-21	AVE
2.58	2.86	2.72

AN-107 Diluted Feed, Sr/TRU and Solids Removal, ICP metals.

recombined waste ug/g		Mass Dilution	Concentration After Treatment DF-01	DF-02	DF-03	DF-04	DF-11		Percent Removed DF-01	DF-02	DF-03	DF-04	DF-11
0	Ag							Ag					
3122.153	Al	1.3075	2140	2230	2220	2270	2220	Al	10	7	7	5	7
2.3205	Ba							Ba					
324.7507	Ca	1.3075	169	160	152	155	157	Ca	32	36	39	38	37
34.50482	Cd	1.3075	26.3	27.4	27.2	27.7	27.1	Cd	0	-4	-3	-5	-3
0	Co	1.3075	2	2	2	2.1	2.1	Co					
138.0323	Cr	1.3075	54.5	7.23	6.65	6.89	51.5	Cr	48	93	94	93	51
0	Cu	1.3075	11.4	12.1	12	12.3	14.8	Cu					
1249.13	Fe	1.3075	11.8	6.49	6.96	7.4	15.9	Fe	99	99	99	99	98
1239	K	1.3075	720	730	716	732	732	K	24	23	24	23	23
5.61	La	1.3075	1.4				1.1	La	67				74
0	Mg							Mg					
329.0137	Mn	1.3075	1.9	15.1	20	17.7	1.8	Mn	99	94	92	93	99
0	Mo	1.3075	14.2	14.8	14.8	15	14.7	Mo					
128028.8	Na	1.3075	112000	116000	116000	118000	119000	Na	-14	-18	-18	-21	-22
287.6623	Ni	1.3075	215	223	222	227	222	Ni	2	-1	-1	-3	-1
217.8798	Pb	1.3075	57.9	59.1	57.5	60.4	65.9	Pb	65	65	65	64	60
0	Si	1.3075	102	43	38	40	46	Si					
0	Ti							Ti					
0	U	1.3075	50	41	39	42	44	U					
3.264	Zn	1.3075	5.7	5.7	5.9	6.4	6	Zn	-128	-128	-136	-156	-140
10.2765	Zr	1.3075	3.8	2.2	2	2.2	3.4	Zr	52	72	75	72	57
	Other Analytes Detected							Other Analytes Detected					
0	As							As					
0	Ce							Ce					
16.2435	Nd	1.3075	6.3	3.1	2.7	2.9	5.1	Nd	49	75	78	77	59
368.2318	P	1.3075	282	293	291	297	294	P	0	-4	-3	-5	-4
2.79	Sr	1.3075	161	90.3	89.9	92.1	153	Sr	-7445	-4132	-4113	-4216	-7070
0	Y	1.3075	2	1.1	0.99	1.1	1.6	Y					

recombined waste ug/g		Mass Dilution	Concentration After Treatment DF-14	DF-20	DF-20 dup	DF-20 ave	DF-21		Percent Removed DF-14	DF-20	DF-20 dup	DF-20 ave	DF-21	Df-20&21 Ave		% removed Average	
0	Ag							Ag							Ag		
3122.153	Al	1.555926	1913	1990	2050	2020	1940	Al	5	1	-2	-1	3	1980	Al	1	0.07644
2.3205	Ba							Ba							Ba		
324.7507	Ca	1.555926	140	140	144	142	131	Ca	33	33	31	32	37	136.5	Ca	35	0.207366
34.50482	Cd	1.555926	22.8	23.4	24	23.7	22.7	Cd	-3	-6	-8	-7	-2	23.2	Cd	-5	-0.002939
0	Co	1.555926	1.9	2.1	2.1	2.1	2	Co						2.05	Co		
138.0323	Cr	1.555926	13.3	37	38.1	37.55	36.5	Cr	85	58	57	58	59	37.025	Cr	58	0.148418
0	Cu	1.555926	13.4	16.3	16.8	16.55	16.3	Cu						16.425	Cu		
1249.13	Fe	1.555926	5.34	9.29	9.16	9.225	8.6	Fe	99	99	99	99	99	8.9125	Fe	99	2.279601
1239	K	1.555926	603	626	634	630	615	K	24	21	20	21	23	622.5	K	22	0.499074
5.61	La	1.555926						La							La		
0	Mg							Mg							Mg		
329.0137	Mn	1.555926	3.9	2.3	2.4	2.35	2.3	Mn	98	99	99	99	99	2.325	Mn	99	0.600499
0	Mo	1.555926	12.4	12.8	13.1	12.95	12.5	Mo						12.725	Mo		
128028.8	Na	1.555926	105000	102000	110000	106000	91900	Na	-28	-24	-34	-29	-12	98950	Na	-20	-47.8523
287.6623	Ni	1.555926	189	194	200	197	187	Ni	-2	-5	-8	-7	-1	192	Ni	-4	-0.020439
217.8798	Pb	1.555926	55.1	54.9	56	55.45	53.9	Pb	61	61	60	60	62	54.675	Pb	61	0.245092
0	Si	1.555926	29	28	25	26.5	32	Si						29.25	Si		
0	Ti							Ti							Ti		
0	U	1.555926	39	39		39		U						39	U		
3.264	Zn	1.555926	5.5	5.7	6	5.85	5	Zn	-162	-172	-186	-179	-138	5.425	Zn	-159	-0.009554
10.2765	Zr	1.555926	1.5	2.1	2.1	2.1	2	Zr	77	68	68	68	70	2.05	Zr	69	0.013078
Other Analytes Detected								Other Analytes Detected							Other Analytes Detected		
0	As							As							As		
0	Ce							Ce							Ce		
16.2435	Nd	1.555926	2.1	2.8	2.7	2.75	2.7	Nd	80	73	74	74	74	2.725	Nd	74	0.022152
368.2318	P	1.555926	246	253	259	256	247	P	-4	-7	-9	-8	-4	251.5	P	-6	-0.042599
2.79	Sr	1.555926	94.4	113	118	115.5	107	Sr	-5164	-6202	-6481	-6341	-5867	111.25	Sr	-6104	-0.31429
0	Y	1.555926		1	1	1	0.99	Y							Y		

Removal of TIC and TOC by Sr/TRU ppt

Recombined Waste	ug/g	Mass Dilution from Sr/TRU Treatment	Concentration After Treatment DF-20 ug/g	DF-21 ug/g
TIC	12284.39	1.555925526	6800.00	6850.00
TOC	22496.04	1.555925526	14000.00	13200.00
TC	34828.89	1.555925526	20800.00	20050.00
TIC	12284.39			

	Percent Removed DF-20	DF-21	AVE ug/g
TIC	13.87%	13.24%	6825.00
TOC	3.17%	8.70%	13600.00
TC	7.08%	10.43%	20425.00
corrected for carbonate removed by Sr ppt			
TIC	6.16%	5.53%	

Matrix	Supernatant				
Lab ID			99-0645	99-0645-d	
Sample ID		AVERAGE	Sample	Duplicate	RPD
Units	Type of Pr	ug/mL (2)	ug/mL (2)	ug/mL (2)	(%)
TIC	Direct/Dilu	16300	16400	16200	1
TOC	Direct/Dilu	29900	30000	29800	0
TC (sum)	Direct/Dilu	46200	46400	46000	1

Centrifuged Wet Solids

		99-0647	99-0647-d	
Type of Pr	AVERAGE ug/g (2)	Sample ug/g (2)	Duplicate ug/g (2)	RPD (%)
Direct	17850	18200	17500	4
Direct	32000	31100	32900	1
Direct	50800	51200	50400	2

Recombined Waste ug/g		Caustic Adjusted Waste ug/g	MW	conc, M
12284.39	TIC	10583.68	12	1.164204
22496.04	TOC	19381.57	12	2.131973
34828.89	TC (sum)	30006.99	12	3.300769

Preliminary results for carbon analyses of Sr/TRU treated AN-107 diluted feed

Lab Number	Matrix	Sampe ID	Hot Cell Dil Factor	diluted sample* Vol ml	Wt g		Total Inorganic Carbon TIC ug/ml	TIC ug/g	RPD (%)	Total Organic Carbon TOC ug/ml	TOC ug/g	RPD (%)	Total Carbon TC ug/ml	TC ug/g	RPD (%)
00-0072 PB	Liquid	Process Blank	6.473	1		0.9862	<6	<6		<17	<17				
00-0072	Liquid	DF-20	8.0167	0.5		0.5099	840	850		1,800	1,800		2,600	2,600	
00-0072 Rep	Liquid	DF-20 Rep	8.0167	1		1.0223	860	880	3	1,800	1,900	5	2,700	2,700	4
00-0073	Liquid	DF-21	8.0192	1		1.0156	850	870		1,600	1,700		2,500	2,500	
00-0073 Rep	Liquid	DF-21 Rep	8.0192	0.5		0.5075	860	870	0	1,600	1,700	0	2,500	2,500	0
00-0074	Liquid	Wash Composite	4.9451	1		0.9857	210	200		480	470		690	680	
00-0074 Rep	Liquid	Wash Composite (Rep)	4.9451	1		0.9905	210	210	5	470	470	0	690	680	0
00-0074 Dup	Liquid	Wash Composite Duplicate	4.9111	1		0.9861	210	210		490	480		700	690	
00-0074 Dup Rep	Liquid	Wash Composite Duplicate (Rep)	4.9111	1		0.9991	220	220	5	510	510	6	720	720	4
99-2350 MS	Liquid	MS Recovery	n/a	0.3			101%			97%			100%		
00-0083	Solid	Washed Solids	n/a	n/a		0.0281	n/a	44,600		n/a	1,100		n/a	45,700	
00-0083 Dup	Solid	Washed Solids Duplicate	n/a	n/a		0.0292	n/a	57,100	25	n/a	1,800	n/a	n/a	58,900	25
00-0083 MS	Solid	Washed Solids MS Recovery	n/a	n/a		0.0286		80%			89%			85%	
00-0083 Rep	Solid	Washed Solids (Rep)	n/a	n/a		0.0306	n/a	51,300		n/a	1,600		n/a	52,900	
00-0083 Dup Rep	Solid	Washed Solids Duplicate (Rep)	n/a	n/a		0.0343	n/a	38,000	30	n/a	2,000	n/a	n/a	40,000	28

							Percent Removed		
		mass diluti	DF-20	DF-21	average		DF-20	DF-21	average
Fluoride	4655.393	1.555926	2000	<2000	<2000	Fluoride	>33%	>33%	<2000
Chloride	976.9118	1.555926	<2000	<2000	<2000	Chloride			<2000
Nitrite (3)	37415.28	1.555926	21800	21600	21700	Nitrite (3)	9.34%	10.18%	21700
Bromide (3)						Bromide (3)			
Nitrate	118005.9	1.555926	86700	88100	87400	Nitrate	-14.32%	-16.16%	87400
Phosphate	2093.382	1.555926	<4000	<4000	<4000	Phosphate			<4000
Sulfate	5695.125	1.555926	<4000	<4000	<4000	Sulfate			<4000
Oxalate (3)	2544.232	1.555926	<4000	<4000	<4000	Oxalate (3)			<4000
							Corrected for addition of $Sr(NO_3)_2$		
Nitrite (3)	37415.28	1.555926	21800	21600	21700	Nitrite (3)	9.34%	10.18%	21700
Bromide (3)						Bromide (3)			
Nitrate	118005.9	1.555926	86700	88100	87400	Nitrate	-6.01%	-7.86%	87400

Date	9/20/99	
Tank	AN-107	NOTES
Filter	Mott 0.1 micron - L	Sheet #1
Test Conditions	Low Solids	
Operator	Ralph Lettau	
Test Engineer	Kriston Brooks	

Test Number	Time	Chiller Temp C	Slurry Temp C	Slurry Loop Flow Rate (gpm)	Filter Outlet Pressure (psig)	Filter Inlet Pressure (psig)	Filtrate Sample Volume (mL)	Time of Collection (Sec)	Filtrate Flow Rate (mL/sec)	Temp.(C)	Filtrate Flux (m3/m2/day)	Permeability (m/day/bar)	Filtrate Flux (gpm/ft2)
1	18:03	25.7	NA	4	48	52	40	45.61	0.877	25.7	4.073	1.181	0.0694
1	18:14	23.6	NA	4.1	47	50	20	46.85	0.427	23.6	2.104	0.629	0.0359
1	18:23	25.2	NA	4	46	50	20	48.17	0.415	25.2	1.956	0.591	0.0333
1	18:33	27.1	NA	4	47	51	20	49.09	0.407	27.1	1.820	0.539	0.0310
1	18:43	24.6	NA	4	46	50	20	61.85	0.323	24.6	1.549	0.468	0.0264
1	18:53	22.9	NA	4	46	50	20	65.35	0.306	22.9	1.538	0.465	0.0262
1	19:03	26.5	NA	4	46	51	20	59.24	0.338	26.5	1.533	0.459	0.0261
										***** Chiller temperature used instead of slurry temper			
2	19:17	23	NA	4.19	26	30	20	44.49	0.450	23	2.253	1.167	0.0384
2	19:27	22.2	NA	4.2	27	30	20	58.7	0.341	22.2	1.747	0.889	0.0298
2	19:39	26.8	NA	4.2	28	32	20	54.52	0.367	26.8	1.652	0.799	0.0282
2	19:47	26	NA	4.2	29	33	20	59.49	0.336	26	1.548	0.724	0.0264
2	19:57	21.9	NA	4.25	28	32.5	20	77.02	0.260	21.9	1.343	0.644	0.0229
2	20:07	23.7	NA	4.2	28	31	20	72.74	0.275	23.7	1.351	0.664	0.0230
2	20:17	27.1	NA	4.3	28	32.5	20	71.81	0.279	27.1	1.244	0.596	0.0212
										***** Chiller temperature used instead of slurry temper			

Backpulsed 2 times

Backpulsed 2 times

Date	9/20/99	Sheet #2
Tank	AN-107	
Filter	Mott 0.1 micron - L	
Test Conditions	Low Solids	
Operator	Ralph Lettau	
Test Engineer	Kriston Brooks	

Test Number	Time	Chiller Temp C	Slurry Temp C	Slurry Loop Flow Rate (gpm)	Filter Outlet Pressure (psig)	Filter Inlet Pressure (psig)	Filtrate Sample Volume (mL)	Time of Collection (Sec)	Filtrate Flow Rate (mL/sec)	Temp.(C)	Filtrate Flux (m3/m2/day)	Permeability (m/day/bar)	Filtrate Flux (gpm/ft2)
3	20:34	22.3	NA	3.3	68	72	40	58.16	0.688	22.3	3.517	0.729	0.0600
3	20:44	27.4	NA	3.4	67.5	70	20	51.55	0.388	27.4	1.718	0.363	0.0293
3	20:55	25.6	24.7	3.5	67	70	20	67.39	0.297	25.6	1.382	0.293	0.0236
3	21:04	24.3	23.2	3.55	66	70	20	77.74	0.257	24.3	1.243	0.265	0.0212
3	21:15	24.7	24.3	3.4	67	71	20	77.27	0.259	24.7	1.236	0.260	0.0211
3	21:24	27.2	26.8	3.45	67	71	20	80.38	0.249	27.2	1.108	0.233	0.0189
3	21:34	25.7	24.8	3.45	66	70	20	88.63	0.226	25.7	1.048	0.224	0.0179
										***** Chiller temperature used instead of slurry temper			
4	21:50	21.7	20.4	3.05	48	50	20	38.15	0.524	20.4	2.832	0.838	0.0483
4	22:01	25.2	24.9	3.05	50	51	20	61.15	0.327	24.9	1.553	0.446	0.0265
4	22:11	27.4	26.8	3.1	48	50	20	71.87	0.278	26.8	1.253	0.371	0.0214
4	22:23	25.6	25	3.15	49	51	20	85.86	0.233	25	1.103	0.320	0.0188
4	22:32	25.1	24.2	3.16	49	51	20	96.45	0.207	24.2	1.005	0.291	0.0171
4	22:46	24.6	24.1	3.15	48	51	20	101.63	0.197	24.1	0.956	0.280	0.0163
4	22:57	25.8	24.8	3.1	48	51	10	54.6	0.183	24.8	0.872	0.256	0.0149

Backpulsed 2 times

Flow began high and droppe within the 1st minute.

Backpulsed 2 times

		Turned on pressure booster	5	23:15	22.6	22.2	4.3	47	51	20	38.06	0.525	22.2	2.695	0.798	0.0459
		Flow & Pressure bouncing	5	23:26	27.7	27.4	4.6	46	50	20	51.56	0.388	27.4	1.718	0.519	0.0293
		P varies by ~ 5-6 psig	5	23:33	26.2	25.4	4.6	47	52	20	58.93	0.339	25.4	1.590	0.466	0.0271
			5	23:43	23.8	22.5	4.5	47	52	20	66.7	0.300	22.5	1.525	0.447	0.0260
			5	23:52	25	24.7	4.5	47	52	20	69.99	0.286	24.7	1.365	0.400	0.0233
			5	0:07	26.2	25.3	4.5	47	52	20	76.02	0.263	25.3	1.236	0.362	0.0211
			5	0:19	23.3	22.2	4.4	48	53	20	88.81	0.225	22.2	1.155	0.332	0.0197
Date	9/21/99	Sheet #3	6	0:40	25.7	24.6	3.97	47	51	20	42.01	0.476	24.6	2.280	0.675	0.0389
Tank	AN-107 DF-Sr/TRU		6	0:50	22.9	21.7	4.02	48	52	20	63.02	0.317	21.7	1.651	0.479	0.0281
Filter	Mott 0.1 micron - L		6	1:00	25.5	25.6	4.15	47	51	20	67.99	0.294	25.6	1.370	0.406	0.0234
Test Conditions	Low Solids		6	1:10	27.1	26.4	4.08	47	53	20	68.2	0.293	26.4	1.336	0.387	0.0228
Operator	Don Rinehart		6	1:20	21.8	23.1	4.05	47	51	20	85.81	0.233	23.1	1.165	0.345	0.0199
Test Engineer	Rich Hallen		6	1:30	24.2	23.7	4.05	48	52	20	84.63	0.236	23.7	1.161	0.337	0.0198
		1:35 - sample collected vial	6	1:43	27.4	26.9	4.1	48	53	20	77.22	0.259	26.9	1.163	0.334	0.0198

Conditions for dewatering are 50 psig and 4.0 gpm
Chiller reset to 27.0 C, waste temp down to 20.8 C

	DW	2:12	26.1	25.7	3.88	47	51	20	50.45	0.396	25.7	1.841	0.545	0.0314
Reset to 26.0 C	DW	2:24	27.9	27.3	4.08	48	53	20	61.03	0.328	27.3	1.456	0.418	0.0248
Sample DF-11	DW	2:33	25.2	24.2	4.03	46	50	20	81.57	0.245	24.2	1.188	0.359	0.0202
Maxed Air Out	DW	2:44	NA	25.4	3.74	45	49	NA	NA		25.4			
Pressure & crossflow down	DW	2:50	NA	29.4	3.62	44	47	20	81.69	0.245	29.4	1.026	0.327	0.0175

* last volume assumed because not entered on data sheet

3:15 CUF shut down and drained

			Test Number	Time	Chiller Temp C	Slurry Temp C	Slurry Loop Flow Rate (gpm)	Filter Outlet Pressure (psig)	Filter Inlet Pressure (psig)	Filtrate Sample Volume (mL)	Time of Collection (Sec)	Filtrate Flow Rate (mL/sec)	Temp. (C)	Filtrate Flux (m3/m2/day)	Permeability (m/day/bar)	Run Time	Filtrate Flux (gpm/ft2)
Date	9/30/99																
Tank	AN-107 Sr/TRU Treated	NOTES															
Filter	Mott 0.1 micron - L	page #1	rerun of 1-	8:08													
Test Condition	Re-constituted waste		rerun of 1-	8:12	26.2	26.8	4.2	45	50	20	62.16	0.322	26.8	1.449	0.442	4	0.025
Operator	Ralph Lettau		rerun of 1-	8:23	26.8	26.5	4.1	46	50	20	70.23	0.285	26.5	1.293	0.391	15	0.022
Test Engineer	Rich Hallen		rerun of 1-	8:34	24	23.3	4.1	46	50	20	84.86	0.236	23.3	1.171	0.354	26	0.020
			rerun of 1-	8:44	26.3	26.6	4.1	46	50	20	84.38	0.237	26.6	1.073	0.324	36	0.018
			rerun of 1-	8:54	27.3	27.2	4.2	46	50	20	80.93	0.247	27.2	1.101	0.333	46	0.019
			rerun of 1-	9:09	24.3	23.5	4.2	46	50	20	94.47	0.212	23.5	1.046	0.316	61	0.018
		Backpulsed 2 times			ave total flow		4.15									ave	0.020
								Ave TMP	47.9167								
Date	9/30/99	Sheet #2	2-1	9:45	26.8	27.2	4	47	51	30	55.59	0.540	27.2	2.404	0.711	0	0.041
Tank	AN-107 Sr/TRU diluted		2-1	9:52	27.2	27.1	4.1	47	49	30	71.09	0.422	27.1	1.885	0.570	7	0.032
Filter	Mott 0.1 micron - L		2-1	10:11	23.7	23	4.1	47	50	20	60.05	0.333	23	1.670	0.499	26	0.028
Test Condition	~25% dilution		2-1	10:24	27.2	27.6	4.1	47	50	20	56.07	0.357	27.6	1.571	0.470	39	0.027
Operator	Ralph Lettau		2-1	10:35	26.6	26.2	4.1	47	50	20	60.62	0.330	26.2	1.511	0.452	50	0.026
Test Engineer	Rich Hallen		2-1	10:46	23.9	23.2	4.1	47	50	20	68.35	0.293	23.2	1.458	0.436	61	0.025
		Backpulsed 2 times - 65 psi			ave total flow		4.083									ave w/o	0.028
								Ave TMP	48.5								
			2-2	10:58	24.3	23.9	4.2	26	30	20	59.73	0.335	23.9	1.636	0.847	0	0.0279
			2-2	11:09	27.1	27.3	4.2	27	30	20	59.23	0.338	27.3	1.500	0.763	11	0.0256
			2-2	11:22	24.8	23.9	4.2	26	30	20	70.81	0.282	23.9	1.380	0.715	24	0.0235
			2-2	11:34	22.6	21.7	4.2	26	30	20	67.56	0.296	21.7	1.540	0.798	36	0.0263
			2-2	11:46	26	26	4.2	26	30	20	70.67	0.283	26	1.303	0.675	48	0.0222
			2-2	11:54	26.9	26.4	4.2	27	30	20	69.66	0.287	26.4	1.308	0.665	56	0.0223
			2-2	12:08	22.6	21.4	4.1	27	30	20	84.39	0.237	21.4	1.244	0.633	70	0.0212
		Backpulsed 2 times			ave total flow		4.186									ave w/o	0.024
								Ave TMP	28.2143								
			2-4	12:23	24.8	24.7	3.1	50	52	20	42.06	0.476	24.7	2.271	0.646	0	0.0387
			2-4	12:32	27.6	27.6	3.1	47	51	20	56.89	0.352	27.6	1.549	0.458	9	0.0264
			2-4	12:44	24	23.1	3.1	47	50	20	73.31	0.273	23.1	1.364	0.408	21	0.0232
			2-4	12:56	23.8	23.4	3.1	47	51	20	78.65	0.254	23.4	1.260	0.373	33	0.0215
			2-4	13:05	26.8	27.1	3.1	48	51	20	75.38	0.265	27.1	1.185	0.347	42	0.0202
			2-4	13:20	24.7	23.9	3.1	46	49	20	89.74	0.223	23.9	1.089	0.332	57	0.0186
			2-4	13:33	23.4	22.9	3.1	46	49	20	95.63	0.209	22.9	1.051	0.321	70	0.0179
		Backpulsed 2 times			ave total flow		3.1									ave w/o	0.021

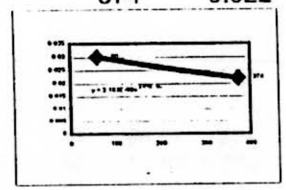
Test 2-Diluted

								Ave TMP	48.8571								
Date	9/30/99	Sheet #3	2-6	13:48	27.9	28.5	4.1	47	51	20	39.69	0.504	28.5	2.165	0.641	0	0.0369
Tank	diluted AN-107 DF-		2-6	14:00	27.3	27	4.1	46	50	20	51.04	0.392	27	1.755	0.530	12	0.0299
Filter	Mott 0.1 micron - L		2-6	14:10	24.8	24	4.06	47	50	20	61.15	0.327	24	1.594	0.477	22	0.0272
Test Condition	diluted		2-6	14:21	24.3	23.9	4	47	51	20	60.91	0.328	23.9	1.604	0.475	33	0.0273
Operator			2-6	14:31	27.4	27.7	4.17	47	51	20	59.43	0.337	27.7	1.478	0.438	43	0.0252
Test Engineer	Rich Hallen		2-6	14:40	26.7	26.4	4.14	47	51	20	63.91	0.313	26.4	1.425	0.422	52	0.0243
			2-6	14:51	24.8	24.2	4.11	46	50	20	72.16	0.277	24.2	1.343	0.406	63	0.0229
		Backpulsed 2 times			ave total flow		4.097									ave w/o	0.026
								Ave TMP	48.6429							2-BP	
			2-7	15:11	26.4	26.8	4.1	47	50	20	45.17	0.443	26.8	1.994	0.596	0	0.0340
			2-7	15:21	28.7	28.9	4.1	47	50	20	46.13	0.434	28.9	1.843	0.551	10	0.0314
		Backpulsed 1 time														1-BP	
			2-7	15:26	27.3	27	4.16	47	50	20	41.91	0.477	27	2.137	0.639	0	0.0364
			2-7	15:35	25.7	25.1	4.11	47	50	20	53.56	0.373	25.1	1.764	0.527	9	0.0301
		Backpulsed 1 time														1-BP	
			2-7	15:40	24.1	23.3	4.07	47	50	20	47.18	0.424	23.3	2.107	0.630	0	0.0359
			2-7	15:51	25.1	25	4.05	47	50	20	57.39	0.348	25	1.651	0.494	11	0.0281
		Backpulsed 1 time														1-BP	
			2-7	15:58	26.6	26.5	4.08	47	50	20	45.02	0.444	26.5	2.018	0.603	0	0.0344
			2-7	16:08	26.8	25.5	4.05	47	50	20	56.56	0.354	25.5	1.651	0.494	10	0.0282
		Backpulsed 2 times														2-BP	
			2-7	16:20	23.9	23.4	4.15	47	51	20	50.5	0.396	23.4	1.963	0.581	0	0.0335
			2-7	16:30	27.3	27.5	4.21	47	51	20	53.31	0.375	27.5	1.657	0.491	10	0.0283
		Backpulsed 2 times							50							2-BP	
			2-7	16:43	25.3	24.6	4.2	46	51	20	48.98	0.408	24.6	1.956	0.585	0	0.0333
			2-7	16:55	24.2	23.4	4.2	47	53	20	61.46	0.325	23.4	1.613	0.468	12	0.0275
		Backpulsed 1 time			ave total flow		4.123									ave all	0.032
								Ave TMP	48.76								
		Conditions for dewatering are 50 psig and 4.0 gpm															
			DW	17:07	24.6	24.5	4	47	52	20	57.38	0.349	24.5	1.674	0.491	0	0.0285
			DW	17:19	28.9	29	4.2	47	51	20	57.81	0.346	29	1.466	0.434	12	0.0250
			DW	17:32	26.2	25.6	4.25	47	51	20	66.65	0.300	25.6	1.398	0.414	25	0.0238
			DW	17:45	24.3	23.4	4.2	47	52	20	77.16	0.259	23.4	1.285	0.376	38	0.0219
			DW	18:04	26.6	26.3	3.48	43	47	20	89.52	0.223	26.3	1.020	0.329	57	0.0174
					ave total flow		4.026									ave w/o	0.022
								Ave TMP	48.4								

time	total time	test #	TMP (psig)	Total Flow (gpm)	Velocity (ft/sec)	Ave Flux (gpm/ft2)
60	60	1	49	4	11.62181	0.03
60	120	2	30	4.2	12.20291	0.025
60	180	3	69	3.4	9.878543	0.022
67	247	4	50	3.1	9.006906	0.019
64	311	5	49	4.5	13.07454	0.024
63	374	6	50	4.1	11.91236	0.022
38	412 DW		48	3.9	11.33127	0.021
				factor	2.905454	

60 0.03 y = 3.183E-02e-9.878E-04x

374 0.022



test #			Ave Flux	% Change
	1	60	0.030	
	2	120	0.028	5.75%
	3	180	0.027	5.75%
	4	247	0.025	6.40%
	5	311	0.023	6.13%
	6	374	0.022	6.03%
DW		412	0.021	3.68%

so a	63.33%	pressure increase =	13.09%	flux increase
and a	29.03%	velocity increase =	31.26%	flux increase
and a	12.50%	velocity increase =	2.52%	flux increase
test #3	17.65%		21.11%	flux increase

APPENDIX E: STAFF AND ROLE/RESPONSIBILITY

Staff Member	Role/Responsibility
Richard Hallen	Scientist/Technical Leader - Sr/TRU Precipitation
Paul Bredt	Scientist/Physical and Rheological Properties
Kriston Brooks	Engineer/CUF System and Solids Removal
Lynette Jagoda	Engineer/CUF System Cleaning
Don Rinehart	Technician/Hot Cell Tests-Sr/TRU PPT/CUF Operation
Ralph Lettau	Technician/Hot Cell Tests-Sr/TRU PPT/CUF Operation
Dave Ortiz	Technician/Hot Cell CUF Operation and Cleaning
Vaughn Hoopes	Technician/Hot Cell CUF cleaning and sample prep.
Mike Mann	Technician/Hot Cell Filtrate Composite
Mac Zumhoff	Technician/Hot Cell Operations

Distribution

No. of Copies

OFFSITE

2 DOE/Office of Scientific and Technical Information

No. of Copies

ONSITE

5 British Nuclear Fuels, Limited

M. E. Johnson (4)	BN-FL
A. Thompson	BN-FL

11 Pacific Northwest National Laboratory

P. R. Bredt	P7-25
K. P. Brooks	K6-24
R. T. Hallen	K2-12
L. K. Jagoda	K6-24
D. E. Kurath	P7-28
E. V. Morrey	P7-28
Technical Report Files (5)	K1-06